The Chemical Age

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Notes and Comments

Solvents in Industry

IN industry to-day there are close upon one hundred organic liquids of definite composition in use as solvents, and possibly another two hundred solvent liquids to which trade names have been given. We have long been familiar with hydrocarbon bodies such as benzene and toluene; alcohols, such as methyl, ethyl and amyl; ketones, like acetone; and esters, naming ethyl acetate and amyl acetate among many others. It is comparatively recently, however, that we have made the aquaintance of tetrahydro- and decahydro-naphthalene, of ethyl and butyl lactates; glycols in great variety; cyclohexanes; dichlorethane, trichlorethylene and other chlorinated products; furfurals and such plasticising solvents as triacetin and tricresylphosphate. Industry, however, has demanded such liquids and the chemist has quickly risen to the occasion by supplying them in the quantity and of the purity demanded. The greatest impetus given to the introduction of these new solvents has been the development of the cellulose lacquer industry, but a wider use of wax, tar, oils, gums, resins and rubber has naturally assisted. Simultaneously there has been a greater need for effecting the recovery of the vapours of such solvents when in use, and it is to this particular phase of industry that the chief features in the present issue of THE CHEMICAL AGE are devoted. The principal adsorbents used in recovery systems are activated carbon and a particular type of porous silica which has received the name of silica gel. The relative merits of each of these systems are dealt with in detail in the articles concerned, whilst the present trend of the solvent-using industries in relation to the demand for non-inflammable solvents is indicated in another outstanding feature.

Chemical Patent Amendments

VALUABLE amendments have been secured in the committee stage of the Patents and Designs Bill as the result of energetic action taken by the Joint Chemical Patents Committee, a committee of the Association of British Chemical manufacturers, on which the Chemical Society, the Institute of Chemistry and the Institution of Chemical Engineers are represented. On publication of the Bill the committee examined it from the point of view of chemistry and the chemical industry, and found there were a number of points of importance requiring amendment. As the result of a memorandum to the Board of Trade, followed by a deputation, the committee has secured at the committee stage of the Bill, which was taken last week, almost all the amendments required. One of the most important of the amendments relates to user patents. The Sargant Committee had recommended in its report that there should be a provision for dealing effectively with the abuse of such patents, whereby the manufacture of non-patented substances was being restricted or entirely prevented in this country; the proposed clause in the Bill did not however achieve the object desired. The amendment which the Joint Chemical Committee has secured will do what is wanted, and will remove a long standing hardship to the British chemical industry.

The section dealing with the remedy in case of groundless threats of legal proceedings has also been amended to bring within the scope of actions for infringement persons who threaten on the basis not only of an accepted application, but on a merely provisional specification or unaccepted complete specification. Another important amendment relates to Section 38A, where the Bill proposed the deletion of the word "special" before "methods or processes of manufacture," and the insertion of the word "particular," whereas on the Sargant Committees' own felt that this was an unsatisfactory proposal as the court which would have to construe the section would feel bound to place some meaning on the word "particular," whereas on the Sargant Committees' own finding there was no such meaning intended. An amendment has been secured which avoids the use of the word "particular" and overcomes the difficulty, and the section will now read as follows:—"Except when prepared or produced by the methods or processes of manufacture particularly described and ascertained or by their obvious chemical equivalents."

The Appeal Tribunal

IMPORTANT amendments have also been secured in regard to the appeal tribunal. The Joint Chemical Committee was anxious that the cost of litigation should be kept down, and viewed with anxiety the wording of the proposal in regard to the appeal tribunal because it was felt that since in certain respects the tribunal was to be regarded as a court of the High Court, there would be an inevitable tendency for costs on a High Court basis to be charged. The committee has secured an amendment which will ensure that as

far as costs are concerned the present atmosphere of appeals to the Law Officer where the costs are of a very nominal character shall be preserved. Another amendment which was secured in this clause of the Bill namely, to give the appeal tribunal power to obtain the technical assisance of an assessor in all cases, will go a long way to providing a highly efficient appeal tribunal for dealing with appeals from the Comptroller. These amendments should be of the greatest assistance to the poor inventor.

These represent only the major amendments which the Joint Chemical Patents Committee has been able to secure by its prompt action. The chemical profession and the chemical industry owe a debt of gratitude to the representatives of the various organisations who have devoted such a large amount of time to the work of the Joint Chemical Patents Committee which has achieved such excellent results. The work of the committee is particularly meritorious because the amendments for which it was responsible were the only amendments accepted at the Committee stage, apart from those proposed by the Government and one amendment relating to designs.

New Knowledge on Enzymes

ENZYMES and their action were the subject of a discussion by the Royal Society at a meeting held last week. The main speakers were Professor Willstätter, of Munich, and Professor Waldschmidt-Leitz, of Prague, who, as heads of independent schools, have made fundamental advances in methods of studying enzymes and in the knowledge these new methods have yielded. Enzymes, it will be recalled, belong to the class of substances whose mere presence induces chemical change in other molecules, while the catalyst itself remains unaltered. Complex examples of catalysis in the non-living world are known, and many of them are now utilised in important industrial processes. Catalysis by enzymes, however, differs from inorganic catalysis in many important respects. An enzyme will usually act on only one or a few closely related chemical compounds, and will refuse to touch any molecule coming outside its own limited range of specificity. Its efficiency is usually so great that extremely small quantities suffice to change relatively enormous amounts of the material acted on. In addition, the quantitative study of rate of enzyme action, under different conditions, yields results unlike any type of inorganic catalysis.. Both Willstätter and Waldschmidt-Leitz have worked out methods for extracting enzymes in a state approaching purity, and in the course of this work they have found that " what were really considered to be single enzymes " are really mixtures whose component enzymes show specificity of a very high order.

Chemical and Metallurgical Corporation

THE necessary resolution to carry into effect the re-organisation scheme recently outlined by the director of the Chemical and Metallurgical Corporation was duly approved at the annual general meeting of the company held in London on Tuesday. In the course of his speech, Dr. E. P. Andreae, who presided, said that prices of the company's products had reached new low levels during the period of world-wide depression, and the output of the works was still restricted to a little more than one-half of its productive capacity. In spite of these handicaps, as well as competition which was rendered more severe by the scarcity of business offered, the company had still been able to hold its own and to show a small, though nevertheless significant, increase in turnover. Actually this increase was 5.5 per cent. over that of 1930. Taking into consideration the fact that the average price per ton of the company's products fell by 9 per cent. during this same period, this means that difficulties have been effectively overcome as they arose, and that the recent re-organisation of the works at Runcorn, careful supervision, and rigid economy have enabled the company to bring their costs down to a greater extent than the fall in prices. The plant at Runcorn, as a whole, is now maintained in such condition that it can be brought to full production in the shortest possible time and without further capital outlay. Any improvement in trade would therefore be immediately reflected in the profit and loss account, and given reasonable capitalisation and anything like normal output the company should be able to earn and distribute profits unusually large for this type of business. This is the outlook, even if the activities of the company are restricted to its present products, but it is stressed that the works and organisation of the Chemical and Metallurgical Corporation are something more than a mere undertaking for the manufacture of a few selected heavy chemicals.

Standardisation of Glassware

INVESTIGATIONS which have been proceeding for several years under the auspices of a joint committee for the standardisation of scientific glassware, representing chemical, medical and other scientific organisations, have indicated clearly that there is a demand for standardisation. Various individual efforts have been made to deal with the problem. The Society of Chemical Industry's Committee on the Standardisation of Laboratory Glassware preceded the formation of the joint committee. The Society of Glass Technology has a Glass Standards Committee, which up to the present has been working on problems relating to the chemical properties and thermal resistance of glass, while some of the British Standards Institution's specifications contain details of glass apparatus. Early this year, according to a report in the journal of the Institute of Chemistry, the joint committee gave careful consideration to possible future arrangements for the standardisation of scientific glassware, and it seemed to be the general opinion that the British Standards Institution, as newly re-organised, should be asked to undertake it, and to collate and publish the work already done, as the committee felt that the time was opportune for centralising all work on standardisation under the auspices of the body formed for that purpose.

Assurances were received from the British Standards Institution that it would be willing to undertake the work. Accordingly the committee communicated with the constituent bodies, and all expressed their agreement with the proposals. The Council of the Institute has, therefore, handed the books and papers concerned with the work of the joint committee to the British Standards Institution, together with the balance of the fund in its keeping, and this Institution has agreed to form a special committee under the Chemical Division, which will, in the first instance, consist largely of the personnel of the joint committee.

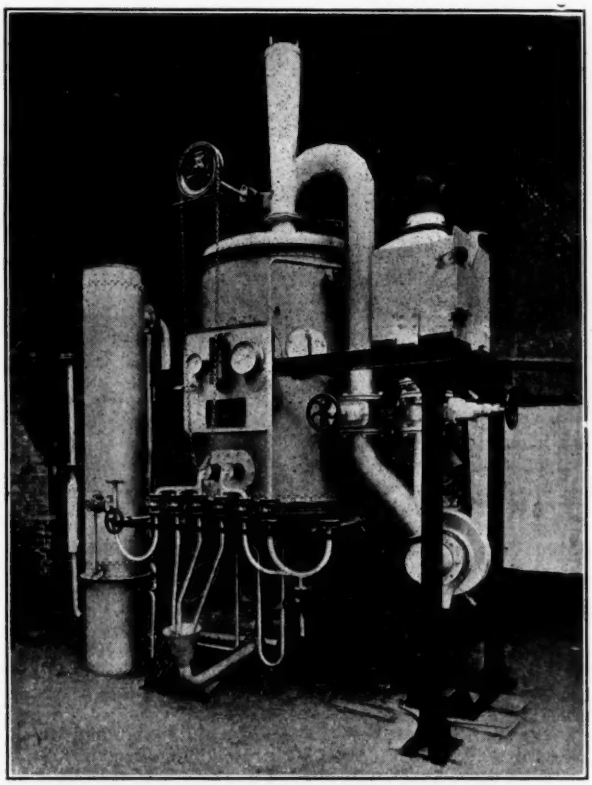
The "Acticarbone" Solvent Recovery Process

By S. REGINALD PRICE

The author of this article is a director of Price, Stutfield and Co., Ltd., London, who are the sole representatives in Great Britain and the Colonies for the "Acticarbone" process.

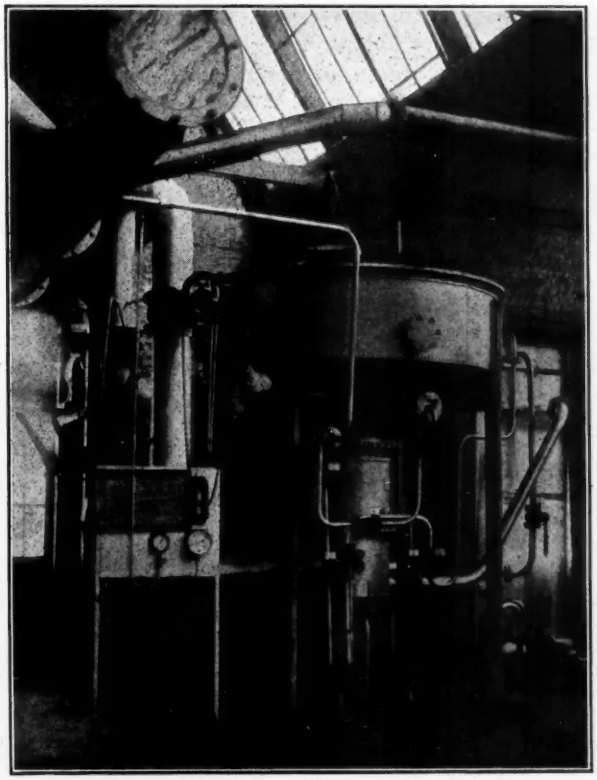
THE principle of solution, with subsequent evaporation of the solvent, is one of the most widely used in industrial processes. Many such processes exist, such as the spreading of rubber solutions on fabrics. waterproofing solutions of other types applied to fabrics, the spreading of the film in the manufacture of imitation leather, the waterproofing of transparent paper, the insulation of paper by varnishing, the dry cleaning of garments, the cleaning and lacquering of metals, and the general application of paints and varnishes. The solvents used in these processes are often costly and their complete loss represents a considerable direct charge on manufacturing costs. Naturally, therefore, considerable attention has been given to means for the recovery of these solvents, and chiefly since 1914 processes have been evolved which allow of a practical and economic solution in many of these recovery problems.

The practical manufacturer interested in this recovery as incidental to his chief operation requires a tested process with guaranteed results backed by wide experience. The capital and operating costs must be reasonable and the process as simple and flexible as possible. All these requirements are fully met by the "Acticarbone" process of solvent recovery elaborated and patented by the Société de Recherches et d'Exploitations Petroliferes, Paris. "Acticarbone" is a highly activated and adsorbent carbon, made from wood by a physical activation process at the Villers-Cotterets factory.



A portable "Acticarbone" Recovery Unit with one adsorber

When a stream of air containing a volatile solvent is passed over this carbon the solvent is almost completely adsorbed even if the concentration is as low as 1 gram. per cu. m. This adsorption continues till the carbon is saturated, when the solvent is liberated by steaming out. The solvent is then condensed and the carbon after cooling is ready to adsorb again. This is in outline the principle of recovery by activated carbon, but the technical application of this principle to give the best and most regular results on an industrial scale has required much patient investigation. As good activated carbon is an expensive material, the correct method of use to ensure a long life of activity is a definite essential of a practical process. The carbon itself can be easily "clogged" or poisoned by certain substances such as sulphur compounds, tarry matters, fine dust, etc., and for best results it should never be wetted in the cycle of distillation.



"Acticarbone" Recovery Plant as erected at works.

The proprietors of the "Acticarbon" process have plants in all parts of Europe which have been working for years on the most diverse problems of solvent recovery and the whole of the experience with these plants is now available for the practical solution of recovery problems. Actual factory results depend on more factors than the efficiency of the recovery system alone. The "Acticarbone" process itself shows an efficiency of recovery of 96 to 97 per cent. or even more, on the solvent vapour entering the plant. The second factor governing efficiency is therefore how much of the solvent vapour evolved during the process can be collected and led to the recovery plant. Here if E represents the percentage efficiency of the recovery plant; e is the percentage efficiency of the collection system for solvent vapour, and P is the percentage practical recovery efficiency, then $P = E \times e$.

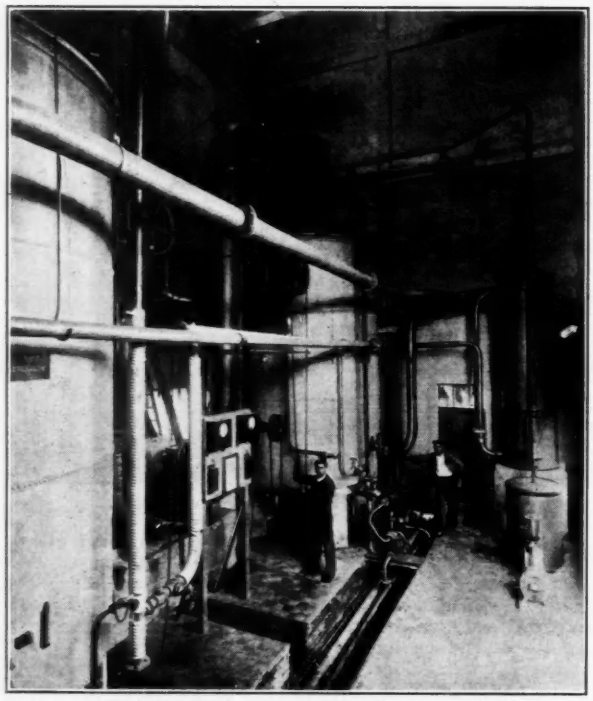
With the "Acticarbone" system E can be 96 to 97 per cent. and the process must be arranged so that this figure remains as constant as possible over a long period. The carbon must also be suitably protected from any materials which interfere with its activity. The efficiency of the collecting system is determined largely by the efficiency of the ventilation system and the direction of the air-solvent stream into the required channels. As the carbon system can operate efficiently at extremely low concentration and large volumes of air

handled, high values for e can therefore be reached in combination with this recovery system. As examples, it may be mentioned that efficient recovery is being carried out from the atmosphere of work rooms and actually from the exits of existing recovery plants working by a washing system. The carbon itself being pure and containing no chemical activators, is quite unaffected by solvents, and these are recovered in a clean and pure state equal in quality to the solvent originally used.

As many of the solvents are inflammable, effective means must be provided to prevent any spread of fire, but here at once we find another of the great advantages of being able to work efficiently at very low concentration. As the vapours can be collected at these low concentrations—much below the explosive limit—the risk of fire is almost eliminated. Recovery by an "Acticarbone" plant is therefore absolutely safe. At the same time, efficient means are taken to isolate any fire that may occur in any section of the collection system, without interfering with the free flow of the air stream.

India-Rubber Works

The solvent to be recovered in india-rubber works is usually a hydrocarbon solvent, such as benzol, petrol naphtha, petrol ether, etc. Wide experience of collection exists for spreading machines, of all types, calenders, cord machines, mixers, dipping plants, etc. As in all collection problems, the system used must not interfere in any way with the process of the factory. For spreading machines, special types of hoods have been designed, combining a high degree of collection, complete safety, isolation and ease of operation. At least



"Acticarbone" Recovery Plant in a Rubber Works: capacity 20 gal. of benzine per hour

35 spreading plants have already been equipped. Calenders with condensation plants can be provided with a supplementary system, so that the overall efficiency on solvent reaches a high figure. The collection from a large dipping factory is obviously difficult, but can definitely be accomplished by this system and efficient plants are in operation. The practical yields in rubber works can vary from 75 to 85 per cent. on spreading machines, to 95 per cent. or so for plants working in closed cycle; for dipping plants the overall yield is somewhat lower. Several plants are operating in this country, and many more are under construction.

In the case of leather cloth manufacture the solvent to be recovered is usually a mixture of alcohol, acetone, esters and hydrocarbons. After recovery these solvents have to be separated and rectified. Methods of collection are similar to those used in the rubber industry from various types of spreading machines. In one large plant in France a mixture of 7 solvents is being dealt with, and including rectification losses, a practical efficiency of over 80 per cent. is being obtained. Others are operating equally successfully and three large plants are at present being erected in this country to deal with such mixtures of organic solvents.

Artificial Silk Manufacture

In this industry the "Acticarbone" process is not suitable for carbon disulphide recovery as the catalytic action of the carbon splits this compound, liberating free sulphur. For the recovery of acetone, ether, alcohol, etc., however, the process is ideal. At present many of the existing factories are already equipped with systems using ice-cold water scrubbing and the efficiency of such plants is usually about 90 per cent. By the addition of a small "Acticarbone" plant to such a recovery system, up to 90 per cent. of the residual 10 per cent. can be recovered, bringing the overall yield of the system up to the figure of 98 to 99 per cent.

The collection problem in a dry cleaning works is naturally rather difficult and to get any good results very large volumes of air must be effectively handled. Filtration of the air stream must also be carefully carried out. In practice the collection of vapours can be made from drying rooms and tumblers, from centrifugals, washing machines and brushing tables. Up to the present fifteen dry cleaning works in France have been equipped with the "Acticarbone" system of recovery and plants are in construction in this country to handle large volumes of air. Trichlorethylene and similar solvents can be recovered as easily as white spirit or solvent naphtha.

In the case of electrical insulating materials, alcohol can be recovered from the process of spreading synthetic resin varnishes on paper or fabric. On account of the action of the volatile compounds in the resins on the carbon, the air stream requires very special treatment. Several plants are operating successfully, giving 75 to 80 per cent. recovery of alcohol.

Solvent Extraction Plants

The economy of a solvent extraction plant for oil, perfume or grease, largely depends on the reduction of the solvent loss to a minimum. The application of the carbon process as supplementary to condensation or oil washing is clear. By leading the exit gases from the condenser or wash tower to a small "Acticarbone" plant, about 95 per cent. of the solvent contained in the exit gas is recovered.

Many other instances of practical results could be given as about 160 plants are in operation or under construction, and with the cumulative experience of these plants, new problems are constantly being investigated and solved. The writer well remembers a process for the crystallisation of T.N.T. from alcohol which was used during the war, the solvent being wasted by evaporation. This was perhaps quite unavoidable in the pressure of the times, but the application of this recovery system to a similar process to-day would result in a recovery of at least 90 per cent. of the alcohol.

Working costs are low, as the requirements of the plant are ordinary steam, cooling water and motive power. About 2d. per gallon of solvent recovered is a good average figure for conditions in this country. The wastage of the carbon is small and due only to the mechanical action of the air current in its passage through the carbon mass. A figure for wastage of 1 to 2 lb. per ton of solvent recovered is general practice. It should also be pointed out that the "Acticarbone" process is useful in improving the working conditions in workshops where volatile solvents are in use. The leakage of solvent vapour with the air is reduced to an absolute minimum which with many solvents means a corresponding benefit to the health of the workers. In some cases the process can be used for the complete deodorisation of the air of work rooms.

Norwegian Gasworks to Recover Benzol

THE Bergen Municipal Gasworks proposes to recover benzol during the process of gas manufacture by adsorption over charcoal. Heretofore, crude tar has been the only by-product recovered, this being sold to refineries in Norway. During 1931, there were produced 12,682,430 cu.m. of gas.

Solvent Recovery by Active Carbon

By J. C. LIDDLE

This article, by the general manager of British Carbo Union, Ltd., London, deals with the advantages to be obtained by the use of active carbon as an adsorbent for solvent vapours. It also features the large benzol recovery plant recently completed at Beckton Gasworks.

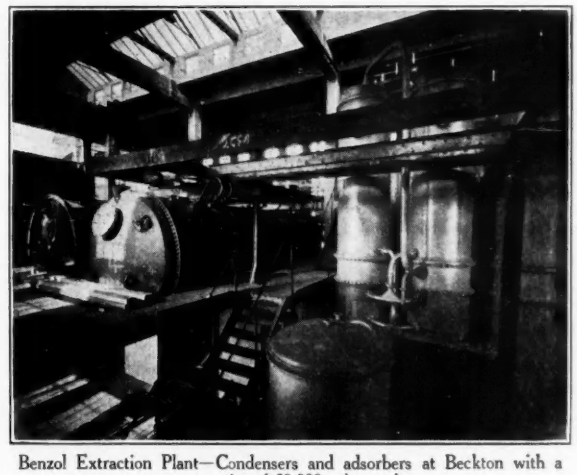
THE system of separating organic vapours from gases by means of active carbon, and the subsequent recovery of the organic compounds by the Bayer steam process, has now been applied commercially for about 12 years. The pioneer work of this process was undertaken by the Carbo Union, who developed it step by step until it is now used with great success in practically all industries where solvent is evaporated in manufacturing processes. This process is also largely used in many oil fields for extracting gasoline entrained in casinghead gas, and it is undoubtedly the most efficient and economical system available when such gas is free, or reasonably free, from H_2S contamination. About 90 per cent of the casinghead gas produced by all of the oil wells in Roumania and Poland is so treated. Plants are also running in British Borneo, Sumatra, Trinidid and the Argentine. The process has also been applied to the extraction of benzol from coal gas, and the largest benzol extraction plant ever built, with a capacity of 20,000 gallons per day, has just been completed at Beckton Gasworks.

Gas Coolers of the new Benzol Plant at the Beckton Works of The Gas Light and Coke Company.

This plant consists essentially of eight large adsorbers arranged four on each side of the centre section, which comprises two steam economisers, four condensers and pre-separators, two main separators, sight boxes and meters. The volume of gas is so large, *viz.*, 75 million cu. ft. per day, that the normal type of vertical adsorber could not be utilised satisfactorily for this purpose and a horizontal design was chosen to give the necessary area. The gas is pumped through the plant by turbine-driven boosters and the exhaust steam from them, at about 5 lb. per sq. in. pressure, is used to supply part of the rinsing steam. The remainder of the rinsing steam is obtained from the economisers, in which a jet of high pressure steam creating a vacuum, causes the condensed water collected from the heating coils to be evaporated, thus supplying a large volume of low pressure steam most economically. The controls for all the valves are brought to the top platform, where eight instrument boards are also situated, one for each adsorber. The valves are at present arranged for operation by hand, but provision has been made for them to be worked by power if this should be found necessary when dealing with full load. The coolers shown in one of the accompanying illustrations are required for reducing the temperature of the gas used for drying and cooling the active carbon. This type of plant takes out more of the lower boiling range hydrocarbons and being more efficient generally, extracts about 20 per cent. more benzol than wash oil. It also removes all of the naphthalene, and most of the CS_2 content from the gas, which is a great advantage from the consumers' point of view. The same system is applicable to quite small gas works and an active carbon plant capable of dealing with a maximum throughput of only one million cubic feet of gas per day is profitable.

Method of Operation

The unqualified success of this system, irrespective of the industry to which it is applied, is due to the remarkable adsorptive properties of active carbon, which enable it to take up a high charge of organic vapours. For instance, of its own weight, a good active carbon will adsorb more than 35 per cent. of benzol at one-tenth saturation. Even at very low concentrations of one-hundredth full saturation, the charge is from 10 to 12 per cent. of its own weight. This property enables the solvent vapour to be mixed with a large volume of air at the points of evaporation, which helps efficient trapping and in the case of inflammable vapours, keeps the mixture well below the lower explosive range, thus practically eliminating fire hazards. When the vapour passes through the carbon, the vapour tension is lowered in its vicinity and in consequence, the vapour in the adsorber will be still less inflammable. When the solvent air mixture is passed through a bed of fresh dry carbon, the adsorption zone occupies only a few inches, and the solvent is extracted in a fraction of a scoend. This action generates heat, raising the temperature of the carbon by about 10° C.



Benzol Extraction Plant—Condensers and adsorbers at Beckton with a capacity of 20,000 gal. per day.

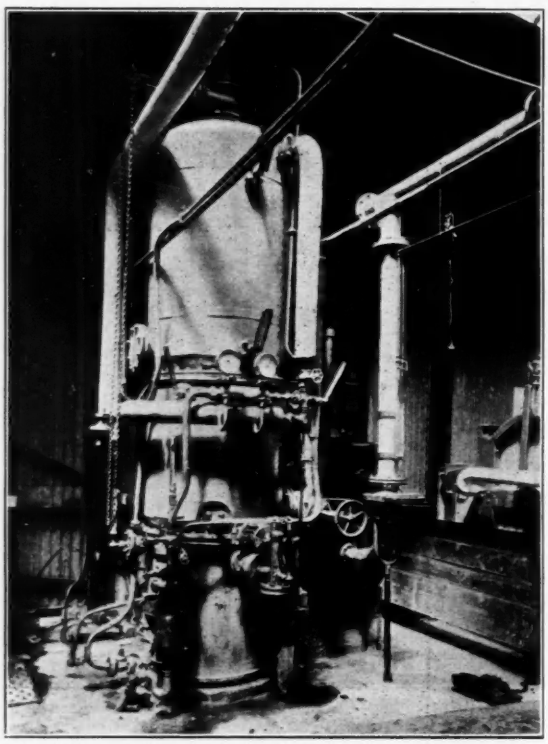
in the adsorption zone, which is used to indicate how the charge is proceeding. As the lower layers become saturated, this zone rises until the carbon is almost fully charged and the solvent vapour is about to break through. This adsorber is then switched out and the gas stream is turned on to another vessel.

The loaded carbon is next regenerated by the direct application of low pressure saturated steam, clean oil-free exhaust steam at 5 lb. per sq. in. being used if available. The weight of rinsing steam consumed is normally about 2½ to 3½ times

the weight of the solvent driven off, so that the condensate will consist of approximately 70 per cent. water and 30 per cent. of solvent. The carbon is now in a moist state, and although it has a selective action for solvent in preference to water, the presence of moisture has the effect of increasing the adsorption zone considerably. If the carbon was used wet, a breakthrough would occur earlier than with dry carbon. It is, therefore, advisable to dry the carbon by hot air or stripped gas, which has been passed through an air heater, consuming approximately 1½ lb. of steam per lb. of recovered solvent. Alternatively some form of heat exchanger may be used. The temperature of the drying air does not usually exceed 100° C. The temperature of the carbon first falls to about 40° C. due to the latent heat of water vapour, and finally rises to about 80° C. when drying is complete. The carbon is, therefore, never subjected to very high temperatures and there is no tendency for oxidation of the solvent to take place. When the solvent is immiscible in water, it separates out from the condensate by gravity but soluble solvents must be separated by fractional distillation. These principles are employed, with modifications in application, in all modern active carbon solvent recovery plants, and this system is now recognised as being the most efficient and economical known method of recovering evaporated solvents, particularly at low concentrations.

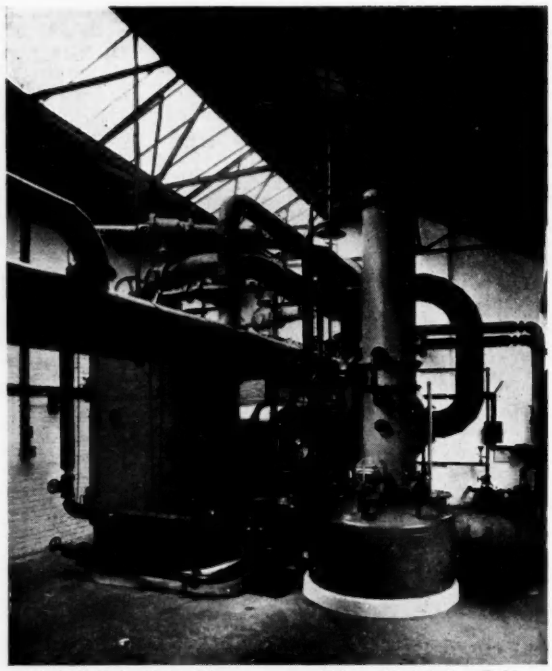
Features of the Active Carbon Employed

The active carbon used by the British Carbo Union is made in cylindrical or rod shape by being extruded through holes during manufacture, forming grains 1/8 to 5/16 in. in length. The most suitable diameter for gas treatment is found to be about 3/16 in., as larger grains would not be so effective on account of the centre mass remaining more or less inert and smaller or irregular grains would cause undue back



Small Recovery Plant for Carbon Tetrachloride dry cleaning—single adsorber superimposed above the condenser and separator.

pressure. This carbon is made tough to withstand the internal pressure strains, to which it is subjected and the shape of the grains gives it further mechanical strength against abrasion and crushing. All active carbons should be removed and sieved to displace fines, at least once every 1 to 1½ years. In a plant working near London, and which was supplied three years ago the carbon was sieved for the first time this year, but only a few pounds of fines passed through ten mesh per inch. Although this loss is negligible, it is still advisable to remove the carbon periodically, as unequal packing and channelling may have developed, causing unequal loads or short circuiting in different parts of the carbon bed. Carbon replacement costs are practically negligible in these solvent recovery plants, and several have completed over 10,000 cycles of operation with the original charge of carbon, which has not become exhausted after recovering over 1,000 lb. of solvent per lb. of carbon.



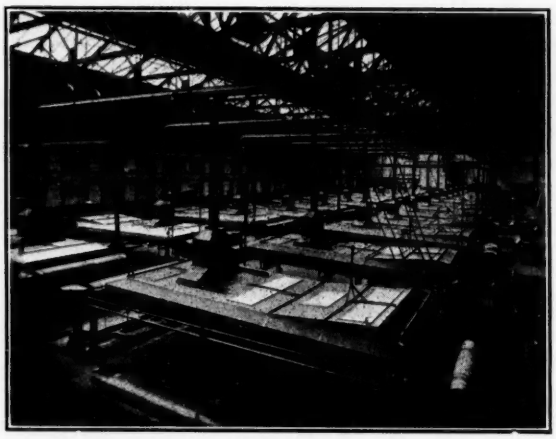
A British Carbo Union Plant recovering Solvent Naphtha in a Rubber Works—capacity 35 gal. per hour.

The high cost of the solvents used in some industries, particularly those connected with the manufacture of nitrocellulose products, would preclude profitable working unless some such highly efficient recovery process were employed. In other industries, where less costly solvents are used, their recovery results in a reduction in direct manufacturing costs and may allow of a better solvent being used to improve the quality of the product or to gain other advantages. In the past, many other systems have been utilised with this purpose in view, but none with the same success. For instance, absorption by liquids, such as water, acids and wash oil have had a certain amount of success when high concentrations were available. The same remarks apply to simple condensation and recovery by compression and cooling, particularly in closed circuits, but in the latter cases there will be no recovery of solvent below the saturation point at the temperature to which it is cooled. In open circuits, weak suction permits escapes, and wide fluctuations are not permissible by these methods. The factory efficiency of such systems usually varies from 40 to 80 per cent. compared with 85 to 99 per cent. obtained by active carbon. Silica gel is also sometimes used as an adsorbent but its adsorptive properties are more adversely affected by variations in temperature than in the case of active carbon; a higher temperature is also necessary for regeneration and it is also more selective to water vapour than to solvent. Where condensation or similar systems are employed, an economical return would usually be shown by connecting up an active carbon plant in series behind such plant, but generally it is preferable to replace them by the more up-to-date process.

Recovery Efficiency and Operating Costs

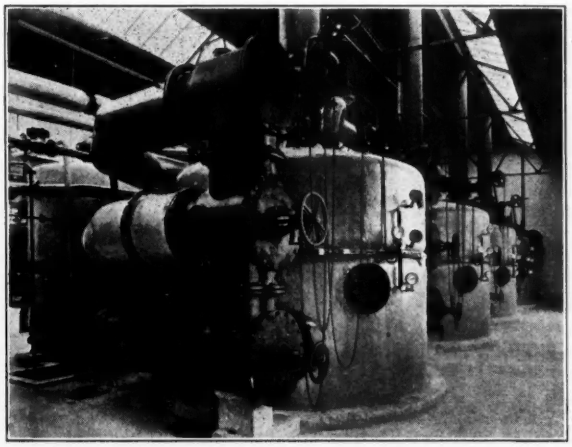
The average losses of solvent through the British Carbo-Union active carbon plants, if properly worked, are only about ½ to 1 per cent., which is negligible. The total factory recovery, therefore, depends on the efficiency of trapping and

on the method of evaporation. In a factory manufacturing "Cellophane" transparent waterproof paper, where a well closed hooding is used, tests carried out about two months ago showed a return of 99.4 per cent. when recovering toluol and ethyl acetate. In the case of an artificial silk works in Lancashire, evaporating over 10 tons of acetone per day, where an active carbon plant was recently installed to replace a water scrubbing plant, there was a recovery last month of 99.5 per cent. In the rubber proofing industry, however, where the hooding is much more open, and where the fabric is not always completely dry when leaving the hooding, and where untrapped losses occur in mixing or from exposure of the dough to atmosphere, a total factory recovery efficiency of over 80 per cent. of solvent naphtha may still be obtained. In factories, where the load remains fairly constant, such as in artificial silk spinning, using alcohol and acetone, a concentration of 25 or 35 grams per cu. m. may be employed, but where the loads fluctuate considerably, as in the rubber industry, an average concentration of 12 to 15 grams is used. These concentrations allow a wide margin of safety and ensure working below the lower combustion range of solvents normally used, which varies from about 48 to 70 grams per cu. m. The strong suction applied at the various points assists evaporation and also ventilates the factory very efficiently and makes conditions more hygienic for the operatives.



A Rubber Proofing Factory at Manchester showing hoodings of British Carbo Union design.

The operating costs of the British Carbo Union plants are very low and may be calculated easily from the following figures, which have been obtained from practical experience with nearly 200 plants. The approximate requirements per ton of solvent are 4 tons of steam, 50 tons of cooling water, 150 kW for the blowers, and 2s. 6d. maintenance charges. These figures do not vary very greatly with the size of the plants, which are very flexible. Overloads of 25 per cent., and temporary peak loads up to 100 per cent., can be dealt with without serious loss of efficiency. When the load is below normal, either the number of vessels in operation may be reduced, or the adsorption period is merely extended, so that steaming out takes place at longer intervals. This explains why operating costs per lb. of solvent recovered remain almost constant under widely varying conditions.



Plant recovering 12-14 tons of acetone per day in a Lancashire Artificial Silk Works—measured efficiency 99.5 per cent. of input.

The accompanying illustrations show some of the plant erected in England by British Carbo Union, Ltd., during the current year.

The Manufacture of Solvents from Esparto Grass

By JAS. MACGREGOR

This article, by the managing director of George Scott and Son (London), Ltd., describes the manufacture of solvents from the non-cellulosic constituents of esparto grass, with the recovery of soda.

Of the vast quantity of esparto used to-day—and the greater part of it in Scotland—about 45 per cent is obtained as pulp. Apart from the waste heat derived from the incineration of the organic matter removed in digestion, and the wax which is recovered on a small scale by one of our most enterprising mills, no value whatever is derived from the balance. A perusal of the patent and technical literature bearing on the subject indicates, however, the vast study which has been devoted to the problem of obtaining useful products from the organic matter present in the black liquor. From the year 1865, in which Routledge was granted a patent for "the recovery of organic substances from the spent or used liquors resulting from the preparation of fibrous substances in the manufacture of paper stock," until the present, many patents have been taken out, but no known process, other than that evolved by Dr. E. L. Rinman—with which this article is concerned—holds any prospect of commercial success.

The Rinman Process

Dr. Rinman's earlier process consisted in mixing the concentrated black liquor with determined proportions of caustic soda and lime and subjecting the resultant mixture to dry distillation. It is not proposed here to deal with the involved chemical reactions which take place. It should, however, be noted that the proportions of caustic soda and lime employed were determined on a scientific basis. This process resulted in very gratifying yields of chemical products, but the chemical engineering difficulties attached to this process lay in the fact that the mixture was a dense plastic mass which could not readily be reduced to a dry state. At the Regensburg mill, in Bavaria, this viscous mass was placed in shallow trays, stacked on racks and carbonised in stationary retorts, similar in construction to those employed on wood distillation plants.

Under Dr. Rinman's most recent patents the use of additional caustic soda is abandoned, and a mixture of barium and calcium oxides employed. When the mixture of these chemicals in a fine state of division is brought into contact with the hot concentrated black liquor a vigorous chemical reaction ensues, resulting in the elimination of the bulk of the water present and the production of a dry powder. This discovery overcame the difficulty attached to the handling of the caustic soda-lime mixture. It also resulted in yields of chemical products never previously attained. At the same time the use of barium introduced new problems. Carbonisation of the mixture of concentrated black liquor, caustic soda and lime, resulted in a hard soda coke. At Regensburg, in the first instance, this soda coke was burned off in a rotary kiln, but this was not entirely successful. Later it was burned on a chain-grate stoker attached to a water-tube boiler,

but the grate area proved inadequate and it was largely burned off in heaps on the floor, as recovered ash is to-day burned off in many esparto mills. The burned residue was leached, the liquor sent to the causticising plant and the carbonate of lime to the lime kiln.

Economies of the Process

The argument may be advanced that paper makers are not chemical manufacturers, but mills engaged in the manufacture of paper from esparto grass are concerned with chemical processes of an involved nature, with which they have been familiar for many years. There is, therefore, no ground for contending that the carbonisation of the black liquor and the production of clean caustic liquor under this process would introduce problems more serious than are attached to the present methods of incineration and causticisation. While in the case of the first plant installed it would be necessary to provide both carbonisation and rectification plants on that site, it is not proposed to attach a rectification plant to each unit. The by-product plant comprises two sections, carbonisation and rectification. At each mill a carbonisation plant would be erected, and from that plant a crude concentrate, approximately 50 per cent., would be delivered to a central rectification plant.

The economies of the process demand that the practically soda-free carbonates be revivified for re-use. That this is feasible and that the revivified chemicals are capable of being re-used in the process without impairing the yields of chemical products, has been fully demonstrated. The practically soda-free wet carbonates mixed with a certain percentage of carbon release the bulk of the admixed water readily. The mixture is calcined at a high temperature in a revolving kiln of the type used on cement plants. The burning of barium carbonate to barium oxide in a revolving kiln in the absence of other bodies has not been achieved, but the presence of calcium carbonate in the process under consideration renders this possible. The water is evaporated and the free carbon is burned out in the back end of the kiln, the decomposition of the carbonates to oxides being achieved in the hot zone at the front end.

In designing the pilot plant, the desirability of doing so on lines which would prove practicable in large-scale operation was kept prominently in view, and in order that the results from this pilot plant might be controlled, parallel tests were conducted in a laboratory plant which permitted of no escape of volatiles by reason of the total absence of moving parts. Designs for a unit capable of dealing with the concentrated black liquor from a mill handling 300 tons of esparto per week are complete, embodying the experience acquired on the pilot plant in so far as the essential features are concerned. It is assumed, however, that the esparto mill will deliver the concentrated black liquor containing approximately 45 per cent. moisture to the by-product plant.

The Carbonising Unit

The mixing of the concentrated liquor with the barium and calcium oxides in the necessary quantities is effected in specially designed steam-jacketed mixers. The steam resulting from the rapid evaporation of the residual moisture in the lye, it is proposed to utilise in the process. The resultant dry brown mass in powdered form is passed to a rotary retort. Here a specially designed steam-jacketed feed hopper and devices for controlling the rate of feed have been evolved. The rotary steel retort is heated externally, the temperature at the inlet end being approximately 220° C. This is gradually increased until at the opposite end, where the carbonised powder is discharged, a temperature of approximately 550° C. is attained. Internally the retort is fitted with breakers and lifting devices, and heating is effected by means of producer gas. When in operation the large volume of hydrogen gas evolved during carbonisation will be utilised for this purpose, but as the chemical reactions in the early stages of carbonisation are exothermic, the heat necessary during these stages is comparatively insignificant.

Carbonisation is effected in a current of super-heated steam of controlled volume and temperature, and the carbonised mass is discharged into a water seal. Leaving the retort, as it does, in a fine state of division and at a high temperature, and containing sufficient calcium hydrate to ensure the desired causticity in the soda liquor, the conversion of the soda from carbonate to hydrate is rapidly effected. The solids are dredged continuously from the sealing vessel, which is suitably designed, into a causticiser. The caustic soda liquor is finally filtered and the filtrate, as already indicated, delivered to the storage tanks serving the esparto digesters. The residue, consisting of barium and calcium carbonates with a certain percentage of free carbon, is washed until practically free from soda, the washings being employed in the sealing vessel.

The Recovery Unit

The gases on leaving the retort enter a dust chamber on the cyclone principle, the temperature in this chamber being maintained at a point which will prevent condensation. From this point, the objects are to fractionate out the heavy oils, effectively trap the ammonia present in the gases, deliver a crude condensate of the maximum concentration and a residual gas free from condensable products. The gases from the dust chamber therefore enter a fractionating or dephlegmating column in which condensation of the heavy oils and a certain portion of the water vapour is effected. From this column the gases pass through a sulphuric acid bath similar to a sulphate of ammonia saturator, the heat of reaction between the ammonia and acid being such as to prevent condensation at this point. The gases then enter the main condenser, where the crude condensate contains the organic solvents which are the object of the process. The gases are thereafter passed through activated carbon, and the adsorbed chemical products are removed from the activated carbon in the usual manner by means of open steam. The gas leaving the adsorbers is chiefly hydrogen, which is exhausted by means of a suitable rotary exhauster and delivered to a gas holder, whilst the watery distillate or crude condensate is passed to the rectifying and fractioning plant, where three columns of standard design are provided.

Yield of Products

The fine products which result from rectification and fractionation consist chiefly of acetone, methyl-alcohol, methyl-ethyl-ketone, iso-propyl-alcohol and di-ethyl-ketone, etc. These have all been isolated and their identity established. Methyl-ethyl-ketone will be marketed as Butanon-1. The group of products recovered from the watery solution, comprising iso-propyl-alcohol, secondary butyl-alcohol, tri-methyl-carbinol and di-ethyl-ketone, will be marketed as Butanon-2. The products which are insoluble in water, consisting of higher ketones, alcohols and hydrocarbons, with a boiling point of 50° C. to 120° C., and practically water white, it is proposed to market as Butanon-3. Derived, as these products are, from a by-product which is at the moment entirely wasted, there should be no difficulty in marketing the output at current prices. The market for methyl-ethyl-ketone is for the moment limited. The invaluable properties of this solvent in the lacquer industry and in the manufacture of aeroplane dope are already widely recognised. The relative yields obtained are:—Acetone 24 per cent; methyl-alcohol 9 per cent.; methyl-ethyl-ketone (Butanon-1) 20 per cent.; Butanon-2 20 per cent.; Butanon-3 25 per cent. The remaining 2 per cent. consists of a very light fraction having a distillation range of 21° C. to 52° C.; this consists of one-third methyl-sulphide and two-thirds hydrocarbons.

Based on a soda recovery of 80 per cent. and on a concentrated black liquor derived from a mill using 20 per cent. of caustic soda, the yield per 100 tons of esparto dealt with will amount to 6 tons. On liquor from a mill using 14 per cent., the yield is rather over 5 tons. The hydrogen gas remaining after the removal of all condensable products, and which in the first instance it is proposed to utilise as fuel, amounts to between 120 and 150 cubic metres per ton of grass.

Brazilian Chemical Organisations

THE Society of Chemists of Rio de Janeiro, established by a governmental decree about six months ago, has recently displayed interest in obtaining foreign contacts. It already has over 100 industrial chemists among its members. It holds semi-monthly meetings for the reading of technical papers and publishes a journal "Revista de Chemica Industrial." It also maintains a library and a permanent committee to assist importers in matters of tariff classification of incoming goods and similar matters. The Society is divided into technical sections according to the industrial interests of members.

Solvent Extraction in the Vegetable Oil Industry

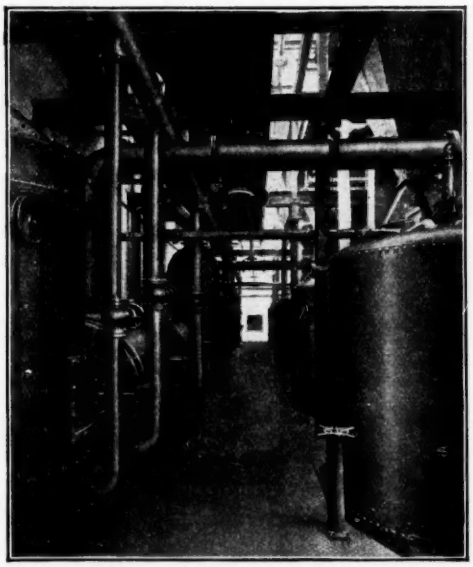
A Résumé of the Process and its Advantages

This article is based on information supplied by Rose, Downs and Thompson, Ltd., of Hull, who have devoted special attention to the design of oil extraction plant for working in hot climates, making due provision for keeping solvent losses down to the minimum obtaining in temperate climates.

IN the field of vegetable oil extraction, solvent extraction and the pressing process each have their would-be positions and spheres of usefulness. Highly efficient pressing machinery for the treatment of oil seeds has been evolved over a period of many hundreds of years, but it is only within the present century that the solvent extraction process has attained its present state of competitive perfection. If it is asked why the solvent process has not to a greater extent ousted its older rival, the answer to this question lies to some extent in the de-centralisation which is taking place in vegetable oil production, whereby the manufacture of the oil is being carried more and more in the countries of origin instead of in the large Europeon and other centres. The solvent process undoubtedly offers its principal advantages in connection with operations on a larger scale and this development, taking place as it has in countries too poorly organised from an industrial and manufacturing point of view, has called for smaller units with a consequent preference for pressing machinery. It is also recognised that the climatic conditions in tropical and sub-tropical countries, which are the largest producers of oil seed, have constituted an obstacle, to the use of the solvent process for which temperate conditions and an ample supply of cooling water are important factors. It might also be remarked that the intensified production of vegetable oils in the large centres during the war has left these with a surplus capacity under post-war conditions which has not yet been absorbed, with the ultimate result that little or no new plant has been put down.

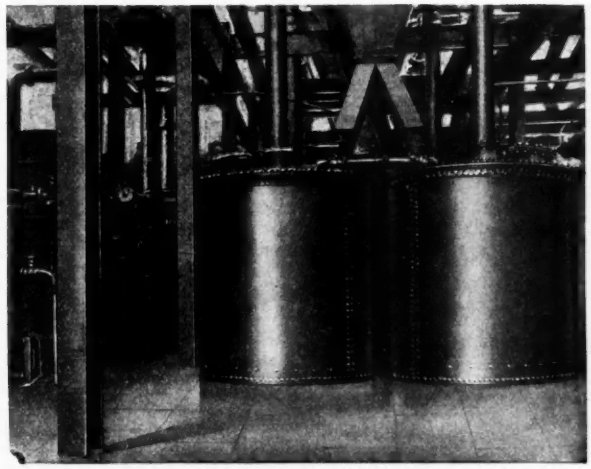
Combination of Processes

A combination of the two processes has been found in some cases to furnish as nearly as possible an ideal installation. In the case of castor seed, for example, it is essential to first press the seed for the production of medicinal cold-drawn oil or high grade lubricating oil, and following this a second treatment by the solvent process enables practically all the remaining oil to be extracted, leaving the residue



Extraction Vessels, Condensers, Evaporators, etc., in a large Vegetable Oil Extraction Plant

almost oil free and in the condition most suitable for use as fertiliser. Such conditions are likely to prevail in the case of any seed, the residue from which cannot be used for feeding purposes, and where it is the primary consideration to extract the maximum possible yield of oil. In the case of materials intended for feeding purposes, it is also becoming recognised more fully that the oil content is not a direct index



Another view of the Extractors and Condensers

to the feeding value beyond a certain point represented by the amount of oil that can be usefully assimilated. There is, therefore, an increasing preference to extract the maximum possible yield of oil, and to adjust the feeding value by the use of other materials such as is done in the manufacture of compound feeding stuffs. Another consideration which has to be taken into account in considering solvent extraction for vegetable oils is that not all seed can be treated with equal facility or with equal results compared with, pressing. Whilst some makers of solvent plant may claim to treat successfully any or all oil seeds, manufacturers recognise that such claims cannot be justified. The suitability of any seed for treatment by the pressing process bears no relation to its suitability for treatment by solvent extraction, and an entirely new technique has been, and is still being, built up as experience is gained with the solvent process. In some cases also, notably that of cottonseed, solvent extraction may affect the oil produced, bringing in its train defects and new problems in the refining of the oil, some of which still remain to be solved.

Advantages of Solvent Extraction

Apart from the vegetable oil production, the solvent process has in many other directions important and extensive particulars, where owing to the nature of the material to be treated, the amount of oil content, and the results required, the pressing process is impracticable. This applies in many cases where the treatment of oil bearing by-products and residues have to be considered; in the extraction of fat from bones, the extraction of fish oils, and the extraction of waxes. In all these cases it is of vital importance to extract the oil content to the lowest possible point, leaving the residue oil free or practically so and the solvent process is the only one which can be considered.

All classes of oil seeds can be treated effectively by the solvent process, and both the oil and the residue can be guaranteed entirely free from any trace of the solvents used. The primary advantage offered by the solvent process is that instead of leaving anything from 5 to 10 per cent. of oil in the residue, as is the case with cakes from a pressing plant, practically the whole of the oil contained in the seed can be removed. The residue from the solvent process, which

is in the form of a meal, may contain as little as ½ per cent. of oil, or in some cases up to 1½ to 2 per cent. Actually, it is possible to extract the whole of the oil; but as the cost of taking out the last traces of oil is more than the value of the oil produced, the process is only carried to what may be termed the economical limit, which varies with different materials between the points stated above. Such results are extremely valuable where manufacturers require to extract the maximum amount of oil or fat.

At one time a good deal of prejudice existed against the use of extracted oils for edible purposes, but to-day large and ever increasing quantities of such oils are employed in the manufacture of margarine and cooking fats, which must necessarily be quite free from any taste or smell of the solvent. It has been stated that extracted oils contains a higher proportion of free fatty acids than pressed oil, but this is not so; the use of the solvent process does not increase the amount of free fatty acid in the oil; the free fatty acid content in both extracted and expressed oils depends entirely upon the quality of the original seed. Again, the extracted meal can now be disposed of quite as readily as the ordinary oil cake, either for feeding purposes or for manure. It is especially suited for the latter purpose on account of the very small proportion of oil it contains, and thus extracted meals which are not suitable for cattle feeding, such as castor, make very valuable fertilisers. In some cases, such extracted meals are mixed with a binding agent, such as molasses, and compressed into feeding cakes or cake nuts.

The Cycle of Operations

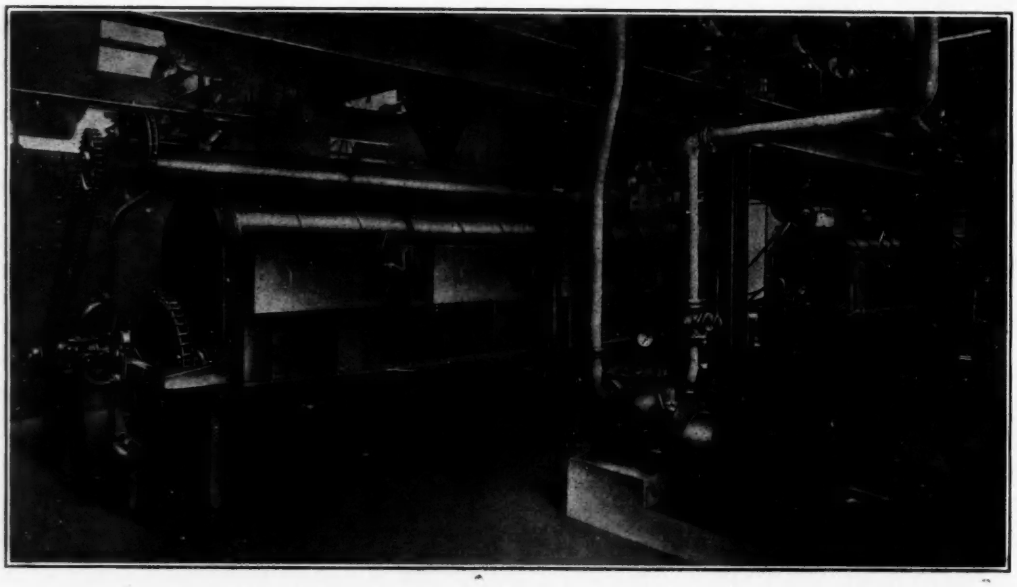
The theory of solvent extraction is that when ground oleaginous seed is saturated with some suitable chemical solvent, the oil is dissolved by the solvent and can be drained off with it. The mixture of solvent and oil is then subjected to heat to evaporate the solvent and separate it from the oil, the vaporised solvent being then condensed and used over again. The process, briefly explained, consists therefore of (1) the preliminary preparation of the seed so that the solvent may easily penetrate the oil cells; (2) the percolation of the solvent (either in the form of cold or hot liquid, or in some cases as a vapour) through the ground seed which is contained in an enclosed pan; (3) the removal of the mixture of oil and solvent to an evaporator, where the solvent is driven off by the application of heat and is recovered by condensation ready to be used again; and (5) the treatment of the residual meal by steam to remove from it any remaining solvent, which also passes to the condensers to be recovered for further use.

The choice of the most suitable solvent will depend upon the circumstances of any particular case. The properties required in an ideal solvent are extremely exacting; it should be neither inflammable nor explosive; it should be of a very searching nature and dissolve the oil content of the material under treatment; it should be a uniform chemical body that will not disintegrate when distilled off, and it should vaporise with the use of only a small amount of heat; it should not give off noxious vapours detrimental to the health of the workmen; it should not cause any chemical change in the material under treatment, nor have any deleterious effect on the plant; and finally, supplies must be readily obtainable at a low cost.

The preliminary preparation of the seed before extraction is of the first importance, and the best results cannot be expected unless this is properly carried out. After cleaning, the seed is ground in practically the same way as for pressing.

From the grinding house the ground material is delivered by means of elevators and conveyors directly to bins situated above the extraction vessels in the extraction house. From these bins the material is charged to the extractors as required, and when charged the vessels are sealed up. The solvent from the store tanks is admitted to the extractor, to which it can be supplied either hot or cold, or as a vapour, as may be required. When the extractor has been charged with solvent, it is allowed to drain down, the mixture of oil and solvent passing to a tank feeding the evaporator. Any mixture of oil and solvent in which the solvent is not fully saturated is passed to a mixed solvent tank, from which it is used for the subsequent first "washing" of a new charge of seed, and by this means all the solvent is used until fully saturated. The charging with solvent and draining down is continued until the drained liquid shows no signs of oil, when the residue remaining in the extractor is thoroughly treated with steam to remove from it all traces of solvent. The discharge door is then opened and the stirrers quickly remove the meal from the vessel, and it passes automatically to the drying machine. After drying and cooling, it is finally dealt with in an automatic sack filling and weighing machine. Where it is desired to reduce the moisture in the extracted meal to a given point, or to control the amount of moisture in the meal within certain limits separate drying machines are also installed.

The oil and solvent from the extractor is treated in the evaporator to vaporise the solvent, which passes to the condensers to be liquefied and returned to the solvent store. The oil is further treated with steam to remove all traces of solvent and is then pumped to the oil store. Special provisions are made to prevent any avoidable loss of solvent, bringing the loss in working down to the absolute minimum, and all the drains and outlets from the plant are carefully trapped to recover any solvent which may be carried off.



Drying Machinery for dealing with Solvent Extracting Oil-bearing Material

Non-Inflammable Solvents

The Chlorine Derivatives of Ethylene and Ethane

This article deals with a range of non-inflammable solvents which are produced on a commercial scale by Imperial Chemical Industries Ltd., and which possess in a marked degree the essential qualities of the ideal solvent.

SINCE 1903 the manufacture of chlorine derivatives of ethane and ethylene, from chlorine and acetylene, has been carried out on a large scale. These chlorine derivatives form a valuable series of non-inflammable solvents whose boiling points range from about 52° C. to 159° C., thus providing useful substitutes for other anhydrous solvents commonly used in extracting oils, fats, waxes, resins, etc., and for dry cleaning, which are inflammable and liable to cause fire and explosion.

Dichlor-, trichlor- and perchlorethylene are stable except when exposed to sunlight, and may be used in contact with all common metals, even in the presence of moisture, although it is advisable to galvanise iron or steel vessels to avoid difficulties due to rusting. Tetrachlor- and pentachlorethane are stable in the absence of moisture under ordinary conditions. In the presence of moisture they are stable in contact with lead and tin, but other common metals reduce them to dichlorethylene and trichlorethylene respectively. The boiling points of the members of this series range from about 52° C. to 159° C., a range the amplitude of which is not approached by any other group of solvents. Their rapidity of penetration is most marked, and the rate of solution of substances is very high, thus permitting very efficient extraction. They are very easily condensed, but dichlorethylene requires a very well cooled condenser on account of its low boiling point. They are non-inflammable, non-combustible and non-explosive, but the hot vapours of dichlorethylene can be ignited and will burn with a cold flame, although this will extinguish itself and there is consequently no risk of fire.

Dichlorethylene and Trichlorethylene

Commercial dichlorethylene does not possess a constant boiling point, since it is a mixture of two dichlorethylenes which are stereo-isomers. One of these boils at 48° C. and the other at 60° C.; the specific gravity of the mixture is about 1.278 at 15° C. Like its relative, trichlorethylene, it is very stable and may be used in contact with all the common industrial metals and even with dilute alkalis. It is an excellent solvent and is now used with marked success in place of low-boiling, highly-inflammable solvents. In the perfume industry it is used for the extraction of delicate perfumes, whose odours would be completely changed by too high a temperature. For rubber, it is the best solvent known. In the laboratory it is also rapidly replacing ether for organic preparations because it is much less dangerous to use, much more easily dried, and the yields obtained are as good as with other solvents, and in some cases better.

Trichlorthylene is a pleasant-smelling liquid of sp. gr. 1.47 and boiling point 87° C. It is the most important member of this group of non-inflammable solvents, and most nearly complies with all the demands made on the ideal solvent. The commercial product is a pure substance, whose boiling point range lies within 1° C. There is no high-boiling residue left behind in the material extracted, so that the last trace of solvent may easily be removed. It does not attack the common metals even in the presence of moisture, and there is no difficulty in constructing plant for its use at a reasonable cost. This solvent is a very rapid worker, and may be used for the extraction of either moist or dry materials. In comparison with benzine, in the same apparatus, it yields a larger output in a given time. It is non-inflamamble, has a low latent heat of evaporation, is heavier than water, and may therefore be kept under water to prevent evaporation.

Uses for Trichlorethylene

Large quantities are used in the extraction industry, where its purity makes possible the extraction of delicate fats without damage. It is also used for degreasing, and for the solution of many resins, tarry and bituminous products, india-rubber and many organic substances, sulphur and phosphorus. It is also used in the preparation of rubber cements and in the varnish, paint and lacquer industry. Its valuable cleansing properties have led to its extensive use for dry cleaning, where its non-inflammability is greatly appreciated; also to its incorporation with soap in laundry and scouring preparations.

Perchlorethylene is a liquid of sp. gr. 1.624 and boiling point 119° C. The pure substance is more expensive than trichlorethylene, but where a solvent is required which is less volatile than trichlorethylene but has the same properties, a mixture of trichlorethylene with 50 per cent. or more per chlorethylene is suitable. Such a mixture does not possess a constant boiling point as is the case with the individual solvents and its composition may change on evaporation, due to the quicker removal of the more volatile trichlorethylene. Tetrachlorethane is the parent substance of the chlorinated derivatives of ethane and ethylene, and is the most powerful solvent of the whole series. It dissolves oils, fats, waxes, resins and tarry substances; also sulphur, phosphorus and chlorine. It dissolves 1 per cent. of sulphur at ordinary temperatures. The solubility increases as the temperature rises, until above the melting point of sulphur the two substances are miscible in all proportion. It is used chiefly as a solvent for varnishes, specially cellulose acetate, and in the preparation of paints; as a paint remover, and to a large extent as a solvent for all kinds of organic products, but on account of its toxic properties it should only be used in specially designed plant.

Oil Extraction by Volatile Solvents

The isolation of animal and vegetable oils and fats is effected by (1) melting either by direct heat of boiling with water or steam; (2) expression; or (3) extraction. In all three processes it is necessary to rupture the walls of the oil-bearing cells. This is done in situ by heating in the first process, whilst for the second and third process, specially where treating seeds and fruit, a preliminary crushing of the material is customary, and is very important if the maximum yield of oil is to be obtained. The process of expression is carried out by means of hydraulic presses. The expressed oil flows away and the residue—press cake—will contain from 4 per cent. to 12 per cent. of oil, which means in many cases a loss of 10 per cent. to 25 per cent. of the original oil content. The process of extraction by a volatile solvent was devised in order to reduce the loss of oil in the press cake by the older process.

The history of extraction of volatile solvents dates back to 1856, when Deiss first used carbon bisulphide for oil extraction in a special plant. In 1864 benzine was first used commercially as a volatile solvent for oils, fats and waxes by Irwine, Richardson and Lundi, and after that the use of volatile hydrocarbons for oil extraction became general, since, in comparison with carbon bisulphide, they are much less inflammable and less explosive, and there is less contamination of the fat. In 1903, when trichlorethylene was first placed on the market, its superiority as a solvent was at once recognised on account of its non-explosive and non-inflammable properties. Since then it has been successfully used for oil extraction, and it is now of great importance in the industry. During this period improvements in its manufacture have been made, and trichlorethylene, as now supplied, is a pure substance which boils over a range of 1° C.

Maximum Efficiency

In order to obtain maximum efficiency in the extraction of solids by trichlorethylene the material to be extracted should be in the form of thin plates, flakes, shavings or threads, in order that the maximum surface may be presented to the solvent together with a minimum thickness of the material. Powdery, sandy, sticky, glue-like or plastic materials are unsuitable for extraction, and should be mixed with an inert substance in order to make the mass more penetrable. Removal of the air from the mass and from the pores of the material is also necessary for rapid extraction, and in some cases it has been found advisable to evacuate the extraction

vessel before admission of the solvent. The design of the plant is also important, as it has been found that where means are provided for reversible circulation of the solvent better extraction takes place, and channelling is avoided. The rate of solution in trichlorethylene increases with the temperature, so that most extractions are carried out with the aid of heat. The most suitable temperature for the extraction of an oil-bearing material can only be found by trial, and by comparing the qualities of the oils produced at the respective temperatures.

In the press process extraction also takes place, the oil itself acting as a solvent which dissolves non-fat impurities, *e.g.*, mucilage and albumen, especially at high pressure and under the influence of heat. In the extraction process where two solvents are dealt with, namely, the trichlorethylene and the oil, the trichlorethylene prevents the extraction by the oil of these non-fat impurities. Extraction consists of diffusion of solvent on the one hand and oil on the other, and as diffusion proceeds more quickly if the solvent is constantly removed, it is better to use small quantities of trichlorethylene often, rather than large quantities less frequently. Generally speaking, it is not economical to remove the last ½ to 2 per cent. of oil from the residue.

Extracted oils are refined in the same way as oils obtained by expression, either by charcoal or silicates. They can now be obtained free from the last traces of trichlorethylene without undue heating and consequent damage, they may be safely used in the manufacture of foodstuffs. The meal residue is also easily freed from the last traces of solvent, and is used in the preparation of compound meals and cattle cakes. Oils may be obtained from castor oil cake, and from fish meal; fats from bones, from the residues obtained in slaughter-houses, and from condemned animal carcases. In these cases extraction gives valuable by-products, and is therefore rapidly superseding the older methods.

Dry Cleaning

Dry cleaning with benzol was introduced in the middle of the nineteenth century, and later petroleum benzine was also used. Non-inflammable solvents are now rapidly displacing both these materials. When open machines are used with inflammable solvents there is continued danger of fire and explosion, even with good ventilation. Enclosed machines are sometimes used to reduce losses and minimise the fire and explosion risks, but at one stage, where the goods, after centrifuging, are transferred to the drying room, the risks are always very great. Such risks can be entirely avoided by the use of trichlorethylene.

The economic advantages of the use of trichlorethylene for dry cleaning, however, are evidenced in more efficient recovery of the solvent and in reduced steam consumption. Goods after cleaning contain a large quantity of solvent, which is removed by centrifuging and final drying in heated chambers operated under reduced pressure. The vapours from the drying rooms are recovered in the most modern plants. The solvent recovered from the washing machines and centrifuges is allowed to settle, and is then filtered in order that the clear solvent containing fat may be separated: in some cases this is used again without purification. Purification of the contaminated solvent is preferably carried out by distillation, either by wet steam or by indirect heating. The former method has the advantage of removing the last trace of solvent from the residue. Where a reasonably dry residue is desired, free also from solvent, the greater part of of the solvent is removed by indirect heating, the last trace being removed by a jet of live steam. Where a constant stream of goods is passing through a dry cleaning works, the distillation residue is obtained in marketable quantity, and may be sold for use in the manufacture of cheap greases and soaps.

Degreasing with Volatile Solvents

Degreasing means the removal of oils and fats from a material, and this is to be distinguished from oil and fat extraction, which has for its primary object the production of oils and fats. Degreasing with volatile solvents is, therefore, a form of dry cleaning applied to articles other than clothing, and oils and fats recovered in the process are regarded as by-products. This process is carried out in the woollen trade, in the tanning industry, in printing establishments, in the electroplating industry, and in works where rags saturated with oils are accumulated in quantity.

In the woollen trade, it is claimed that by degreasing raw wool with a volatile solvent such as trichlorethylene there is a more perfect cleansing of the fibre, the wool fat is recovered in a pure state, the potash salts are more easily recovered, and all subsequent operations in the manufacture of woollen goods are improved, giving a better feel, appearance and durability. The degreasing of woollen piece goods gives a better finish than that secured by the scouring process, with a great saving in soap, and prevents the growth of mildew. In the tanning industries difficulties are encountered with hides that contain an excess of natural fat in isolated spots. This causes staining and uneveness in the feel of the leather, whilst the bad spots must be cut out and sold as low-grade leather. By degreasing the hides, these difficulties are obviated and fats are recovered. Such a process is applied particularly to light hides from sheep, goats, lambs and kids.

In the engineering trade cotton and wool waste and rag of various kinds are used in large quantities by mechanics for absorbing oil. Formerly the dirty waste and rags were burnt, but trichlorethylene is now used for degreasing these cleaning materials, which are thus made fit for further use, and the oil recovered. Considerable economy is thereby effected in large enginering workshops, such as those of the railway companies.

Textile Scouring

Textile materials contain natural oils, fats and waxes, and sometimes added oils, whilst they are occasionally stained with mineral oil. These oils cause the adherance of dirt and grit to the fibres so that the removal of the oils results in a thorough cleansing. The process of scouring consists in the saponification and emulsification of the oils, fats and waxes in the fibre by means of dilute alkalis. Cotton goods are generally given a preliminary scour with milk of lime, but the final scouring is effected with caustic soda, soda ash or silicate of soda, either alone or with soaps. It has been found, however, that the detergent action of soaps is enhanced by using solvents in conjunction with them, but it is essential that the solvents should possess great powers of penetration, and it is advisable that they should also be non-inflamable. These conditions are fulfilled by trichlorethylene which is non-inflammable and is a most powerful solvent, and whose superiority for this purpose is now generally acknowledged.

In recent times, soaps in which trichlorethylene is incorporated have achieved a considerable importance as solvent scourers. These solvent scourers are of two types: those soluble in water, such as "Westropol" and "Westrol," and those which form stable emulsions with water, such as "Westoran." Both types contain neutral soaps and are used for the scouring of textiles of all kinds. As solvent scourers these soaps are most efficient for the scouring of cotton, linen, jute and woollen goods. They will remove oils, fat and waxes, and even the obstinate stains of tar and paint. Even when staining, due to the precipitation of lime and magnesium soaps in the fibre, has been causing trouble, it may be obviated by the use of any one of these products, which dissolve and emulsify the metallic soaps. In the case of woollens felting is prevented, the scoured goods bleach whiter, and brighter colours are obtained by the dyer. For the scouring of cotton, linen, and jute, "Westoran" and "Westrol" are the most suitable, and it is customary to use them in the final sour in conjunction with caustic soda, soda ash or sodium silicate. They may, however, be used for all the scouring operations, in which case the lime boil for cotton goods must be omitted. "Westropol," with an olive oil base, is used more particularly for the scouring of fine woollens, including hosiery and woollen underwear. By its means a better feel and appearance of the goods is secured, and subsequent bleaching and dyeing give superior results.

Sulphuric Acid in Yugoslavia

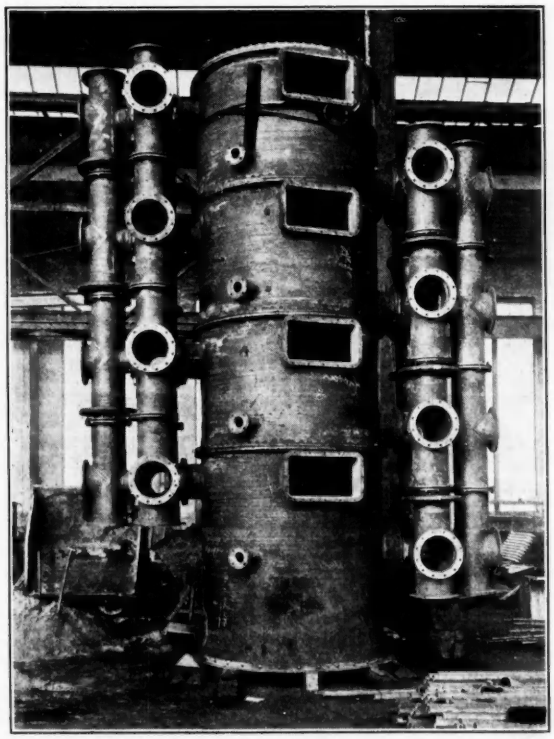
YUGOSLAVIA'S production of sulphuric acid of all kinds in 1931 was approximately 12,000 tons. About 450 tons of sulphuric acid were imported into Yugoslavia during the year 1930 and about 60 tons exported. Of the imported acid, which was mostly concentrated acid, Austria supplied the major portion. The four oil refineries in the country consume 600 to 900 tons of sulphuric acid per year.

Solvent Recovery by Silica Gel

An Outline of its Salient Features

AMONG the various methods employed for the recovery of volatile solvents silica gel has been recognised as having many sterling qualities from the points of view of ease and safety of operation, long life and high adsorption capacity. Ease and safety in operation removes from the users of the silica gel process of solvent recovery the necessity of supervision by skilled labour, ensures a minimum of attention being required and therefore low running cost and the freedom from anxiety of explosion risks and fire. The life of silica gel is ensured by the hardness and inertness of its granules coupled with the complete reversibility in adsorption and desorption and perfect adaptability for the recovery of volatile solvents. Its high adsorption capacity enables plants to be designed for installation in very restricted space and adds to the low cost of operation. In many cases plants may be installed actually in the process room, thus avoiding the necessity of a separate building and excessive duct work in which loss may take place.

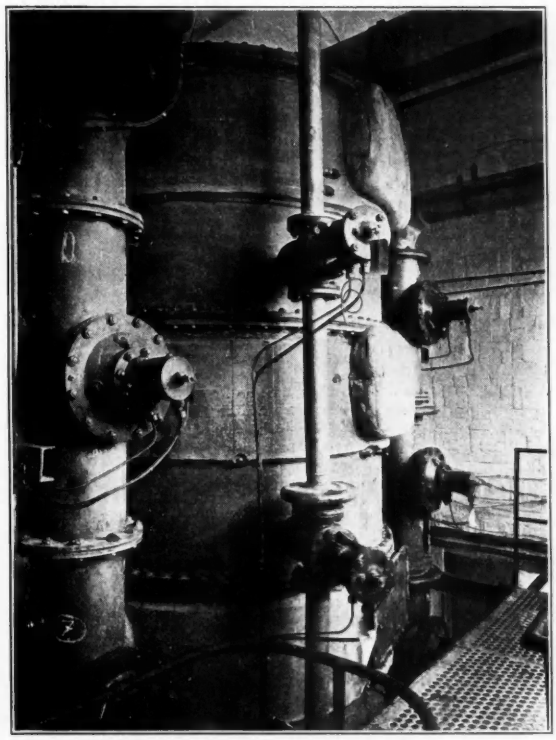
The silica gel process, whether it is a question of recovering alcohol, ether, acetone, or petrol is essentially the same. The number of adsorbers may be two, but in the majority of plants there are four. The adsorbers, generally of rectangular cross section, but in some cases circular, are placed above one another with ducts and valve connections at two opposite sides in one compact assembly, thus requiring a minimum of floor space. The operation of the plant is entirely automatic and needs only the usual occasional attention for the purpose of ascertaining the correct performance



Silica Gel Solvent Recovery Plant (circular type) under construction

of automatic equipment. The solvent laden air flows through two adsorbers in series for a certain length of time (called a period), depending on the nature of the solvent and its concentration in the air. Before entering the second adsorber the air is generally cooled in order to remove the heat of adsorption and thus ensure as complete a clean-up as possible. By this arrangement fluctuations in concentration and overloads of short duration are automatically taken care of. From the second adsorber the stripped air flows to the atmosphere or part of it may be returned to the process room if desirable.

While two of the adsorbers are adsorbing, the third is being desorbed. Superheated steam flows into and through the gel bed, evaporates and displaces the solvent which it carries to the condenser. The steam consumption is low, but varies somewhat with the characteristics of the solvent, being three weight units of steam per weight unit of solvent miscible with water and less for solvents not miscible with water. The steam temperatures are generally carried as high as possible without causing breakdown of the



Silica Gel Solvent Recovery Plant in operation

solvent, since high temperatures mean low steam consumption and overall saving of heat. The fourth adsorber is at the same time being activated; that is, the water which the gel adsorbed during desorption is being driven out by means of a stream or hot air. The heat source for this purpose may be high pressure steam, a combination of exhaust steam and high pressure steam, waste heat or any sort of fuel available. At the end of each period the automatic control changes the position of the valves controlling the flow of steam and air streams in such a manner that all the adsorbers now enter another phase in their path through the complete cycle, first stage adsorption, desorption, activation and second stage adsorption.

The process as described gives very high recovery yields, and all plants are sold under a guarantee to recover a certain fixed percentage of solvent. A recent test made on an acetone recovery plant in Switzerland was conducted on the following lines in order to obtain accurate and indisputable data. A weighed quantity of acetone was evaporated into an air stream entering the plant at a rate corresponding to the concentration under which the plant normally operated and the amount of solvent recovered in the tank collecting the condensate from the condenser was determined by analysis. This test gave a recovery of 96 per cent., while the guarantee given with the plant was 92 per cent.

The foregoing notes give the salient features of silica gel, and should convince those who have recovery problems that this material is an efficient one, fully deserving their investigation and consideration.

Dammar for Nitro-Cellulose Lacquer Manufacture

The Importance of Proper Dewaxing

CREX dammar, a specially prepared " dewaxed " gum is the subject of an addition to the interesting series of booklets on modern lacquer, issued by Rex Campbell and Co., Ltd., 7 Idol Lane, London, E.C.3. The superiority of this product rests on the fact that it is prepared only from selected grades of dammar, which have proved over a considerable period to be suitable for blending, and deliveries of which have been found to be constant. Dammar from such species of tree as *Hopea* or *Balonocarpus* is first sorted, matched and subjected to rigid tests to ensure absolute uniformity of colour and gum properties. After the removal of the last traces of dirt and extraneous matter, the grades chosen are then blended to meet the requirements of the lacquer to be produced. The hard " beta " resins (which have come to be spoken of as " wax ") are removed and the gum is packed in lumps in cases ready for immediate addition to the mixing drum in any quantity at any time. This careful selection, blending and scientific treatment enables each delivery to be uniform, and in the manufacture of nitro-cellulose lacquer the use of Crex dammar therefore implies sound lacquer formulation and ensures accuracy throughout the entire process of production.

Ill-Effects of " Home Dewaxing "

It has been claimed that a better product is obtained when the individual user undertakes his own dewaxing. It is true that a pale gum can be obtained by " home dewaxing " some times, but the batches are subject to variation in colour and other respects (of which the lacquer-maker is usually not aware) due to the differing types and qualities of natural gum on the market. The fact that ordinary dammar is a natural product should not be lost sight of. The gum, derived from trees of many species growing in various districts in several parts of the world and under different climatic conditions, comes to the industrial user just as it is shipped. Every parcel, therefore, differs in some respect, and often in one or other essential characteristic—colour, melting point, solubility, so that the lacquer-maker cannot be expected to know for certain the results he will produce. Moreover, detection of variation in the quality of deliveries is difficult except by an expert in gums.

As nitro-cellulose cottons, solvents and diluents are supplied to standard specifications, the lacquer chemist knows exactly what results he will obtain from these ingredients. With ordinary gums and resins no such guarantee of specification is given; every consignment must be purchased on its merits. Ordinary gums and resins (excepting some types of synthetic and esterified products) are the unknown factors in modern nitro-cellulose lacquer production; and this is especially true of dammar, whether natural or " home dewaxed." Moreover, in the process of dewaxing by the individual industrial user there is present in the final product a quantity of alcohol in addition to diluent which in some formulæ is not required. Unless several different batches are prepared—involving extra work and costs—strict adherance to such formulæ is impossible. " Home dewaxed " dammar is, therefore, unscientific, for chance plays too large a part in the process. The least that can be said is that accuracy is not maintained throughout production; nor is the best use made of formulæ, since the ultimate value of these must always be measured by the least satisfactory ingredient. Many reasons have been given for blushing. Not the least important of these is the use of unsatisfactory gum. " Gum blush " is partly due to incompatible or inferior gums; " orange peel " arises largely from the same cause. These defects are all accentuated with the use of " home dewaxed " dammar, whether prepared in large or small quantities.

Chemical and Physical Characteristics

The colour of Crex dammar is light pale amber. Its melting point is 80-81° C., ash content, 0.013 per cent.; acid value, 27.95; saponification value, 38.50. It is completely soluble in amyl alcohol, amyl acetate, butyl alcohol,, butyl acetate, ethyl acetate, ethyl lactate, xylol, toluol, benzol and other lacquer solvents and diluents, but is insoluble in methyl alcohol.

In regard to the comparative prices of this scientifically dewaxed dammar and " home dewaxed " gum, there are in the preparation of the latter a number of invisible costs which are usually overlooked. Dirt and extraneous matter in each delivery averages five per cent. of the bulk. Loss of gum in the form of " wax " accounts for approximately 20 per cent. Evaporation of solvents in dissolving and filtration increases the cost by another 10 per cent., while the cost of the time taken to do the work claims an additional percentage. Nor should it be overlooked that a factory operation is involved in the process. When this invisible costs of " home dewaxing " are calculated into the price, Crex dammar is by no means dearer. On the contrary, the fact that it eliminates one complete factory operation—involving a troublesome process no longer regarded as the work of the lacquer chemist—has shown it to be a cheaper and more convenient product.

The Newer Solvents for the Solvent-Using Industries

Cyclohexanol and Some Related Products

Howards and Sons, Ltd., of Ilford, were the pioneers in manufacturing the solvents dealt with in this article, and in nearly every instance they remain the sole British producers.

A LARGE number of solvents of British manufacture throughout, are now available, which in quality and price give the consumer a considerable advantage over similar products of foreign origin. A typical example is the cyclohexanol series of solvents and plasticisers manufactured by Howards and Sons, Ltd., of Ilford. This range includes cyclohexanol. methylcyclohexanol (Sextol), cyclohexanone (Sextone), methylcyclohexanone (Sextone B), cyclohexanol acetate, methylcyclohexanol oxalate (Barkite) and cyclohexane. The most striking feature about this series of solvents is the large variety of purposes for which they are used: this is due especially to their very high solvent powers for an exceptionally wide range of susbtances, together with the fact that nearly all of them posses very high flash points, great stability and entire absence of toxic and corrosive properties. The quality of each member of this series is being constantly improved as the result of continuous research on manufacturing processes.

These solvents are used in the following trades, to mention the most important ones: Cellulose lacquer, synthetic resin lacquer, film, artificial leather, plastics, paint and varnish, rubber, waterproofing, polish, electrical, wireless, engineering, leather, textile, laundry and dry cleaning. In the cellulose lacquer trade the ketones and esters of this series are valuable on account of their high solvent powers for cellulose esters and ethers, a very wide range of natural and synthetic resins, oils and dyes, etc. They impart good flow, gloss and resistance to blushing, the ketones have very high dilution ratios, and both ketones and esters possess useful blending powers for cotton and resins and cotton and oils; the oxalates, besides possesing good plasticising properties, have a very high resistance to ultra-violet light. One of the most outstanding features in the lacquer and leather-cloth trades is the great increase in the quantities of Sextone and Sextone B that have been used in the last two or three years and there are obvious signs that their popularity is likely to increase still further. In addition to their uses in the above trades the ketones and acetates of the cyclohexanol series find considerable application in high flash-point paint removing compositions and in the degreasing of metals and leather.

They are very good solvents for waxes. Their high solvent powers for rubber, coupled with their high-flash-points and non-toxicity, make them of considerable interest to this trade in spite of their difference in price over the common rubber solvents. The alcohols of this series are also largely used in synthetic resin lacquers (of the stoving type), paint removers, and for degreasing metals and leather.

Cyclohexane (fully hydrogenated benzene) shares with the other members of this group the property of being an excellent solvent for a large number of substances but differs from them in being a low-boiling solvent. It is a particularly useful solvent for recrystallisation purposes in the fine chemical industry on account of its sharp boiling-point, pleasant odour and low price, and has these great advantages over the special petrol cuts which are otherwise used; for the same reasons it is also a valuable solvent for use in the extraction of essential oils. Cyclohexane is one of the most powerful rubber solvents known and an excellent solvent for waxes, especially paraffin wax, oils and fats.

Cyclohexanol and Sextol

The alcohols of this series, cyclohexanol and Sextol, are used to a very large extent in the textile, dry-cleaning and laundry trades to assist scouring, degreasing and cleaning, etc. They are excellent solvents for oils, fats, waxes, paints, tar, pitch, resins, etc., being particularly efficient solvents for mineral oil and oxidised oils. They have good wetting-out power and they can very easily be incorporated in soaps for use in these three trades, giving products having much higher detergent powers than the ordinary soaps used; alternatively they may be added to the soap liquor in a stock tank or to the dilute soap or soda-ash liquor in a scouring or dyeing bath. These two solvents, in addition to the great ease with which they are incorporated in soaps, which give clear solutions with water, possess the further advantages of having a very high flash-point, being non-toxic and non-corrosive, and being very stable. Some of the principle uses of Sextol and cyclohexanol in the textile trade are in the scouring of raw wool, yarns and woollen and worsted pieces, in the kier-boiling of cotton to assist both scouring and penetration, in the scouring of hosiery, for silk de-gumming, for desizing artificial silk and for removing oil-stains from artificial silk and real silk pieces, for assisting levelling and penetration in dyeing, and in the preparation of spinning oils for woollens and worsteds, lubricants for artificial silk hosiery, and finishing emulsions for silk and artificial silk. A fairly recent use of Sextol in the textile trade is in the delustreing of acetate artificial silk. For laundry work these two solvents are also being used on overalls, woollens, artificial silks and difficult jobs such as cuffs and collars.

For dry-cleaning, Sextol soaps possess the advantages over the ordinary dry-cleaning soaps of much improved solubility, stability and cleaning power; in addition, Sextol soaps have very high water-carrying powers; they give excellent results when used for spotting purposes. Both in the laundry and dry cleaning trades the high flash point, non-toxicity and non-corrosive properties of these solvents, combined with their great stability, make them particularly suitable for use in these trades.

Ethyl Lactate and Diacetone Alcohol

Two other interesting solvents, whose manufacture and use in this country have both made rapid strides during the last few years, are ethyl lactate and diacetone alcohol. Here again both the quality and price in each case gives the consumer a big advantage over the foreign-made product. The quality of the ethyl lactate now on the market shows a very great improvement over that of about three years ago and the use of this solvent has rapidly increased during the last twelve to eighteen months. There has recently been put on the market a quality of diacetone alcohol far in excess of any hitherto produced, both as regards freedom from impurities and stability generally, and there is no doubt that the already expanding use of this solvent will show a considerable further increase now that this greatly improved quality is available. Ethyl lactate and diacetone alcohol are used in the cellulose lacquer, film, artificial leather, safety-glass, paint and varnish, and textile trades principally. In the cellulose lacquer trade and in the leather-cloth trade the use of ethyl lactate has increased enormously during the last eighteen months. It is an excellent solvent for both nitro-cellulose and cellulose acetate, imparts good flow to lacquers, has strong anti-blushing properties, a very high dilution ratio and a mild and pleasant odour. It is also used as a high-boiling solvent in french polishes of the spraying type. Diacetone alcohol is a useful solvent for brushing lacquers, acetate dopes and films, and acetate safety-glass manufacture.

Methyl ethyl ketone, of British manufacture, is now available in this country at a price substantially below that of the imported product. It is used in the manufacture of acetate dopes, cellulose lacquers, etc. Another solvent of considerable interest which is also now on the market and of British manufacture is dipentene (100 per cent. *dl.* limonene). It has a high flash-point, pleasant terpene smell and is an excellent solvent for all vegetable oils and natural resins, also for many synthetic resins. Dipentene is rapidly being taken up by the paint and varnish trade as it is more efficient than turpentine in its various applications and considerably cheaper.

Bleachers, Dyers and Finishers

New Federation of Operatives' Unions

THE Federation of Bleachers, Dyers, Finishers and Calico Printers was constituted on June 18 at a conference of trade union executive committees at Bradford. The unions concerned are the Amalgamated Society of Dyers, the National Union of Textile Workers, the Operative Bleachers, Dyers, and Finishers (Bolton Amalgamation), the National Union of General and Municipal Workers, and the Calendermen's Society, whose members engaged in the trades covered number about 70,000.

According to a statement issued by Mr. Arthur Shaw, secretary, the object of the Federation, which takes the place of the old Dyers' Joint Council—a more or less informal organisation—are to assist the affiliated unions, to promote amalgamation among them, and "to secure co-ordination and solidarity in every effort to raise the wages and improve the conditions of members of affiliated unions." In the case of a dispute arising the societies are to carry out the instructions of an executive committee, subject to confirmation by a general council which is to be the governing body, composed of members of the executive committees of the union and the officers.

Resolutions were brought forward at the conference on payments for holidays and a general advance in wages, but, Mr. Shaw stated, it was felt that the time was inopportune to make applications. It was decided, with the object of alleviating unemployment, to make application to the Allied Association (the employers' organisation) for a limitation of overtime to 4½ hours per week in excess of the 48-hour working week. The Federation was disposed to consider the establishment of two and three shift systems. It was uncommon to work three shifts and there might be opposition from trade union members unaccustomed to these, and in particular from those who were working in excess of 48 hours. It had been decided also to ask the unions to prepare a schedule showing in what occupations in the industry women had superseded men during the past ten years.

A resolution of protest against the means test was passed, which called for the authorisation of all unemployment benefits by officers of the Ministry to secure uniformity in administration.

New Cyanamide Plant in Italy

THE electro-chemical plant of the "Terni" company at Papigno was recently completed. It is reported to contain the most modern equipment for the manufacture of calcium carbide and cyanamide. The capacity is far in excess of the present Italian demand but the promoters anticipate that Italian consumption of nitrogenous fertilisers must increase with the development in growth of Italian agriculture. Cyanamide production of the Terni company amounted in 1931 to 46,762 metric tons, as compared with 65,183 in the preceding year. Calcium carbide sales were 10,070 tons and 11,500 tons, respectively. It is planned to limit production of ammonium sulphate at the Terni's Nera Montoro plant to 28,000 metric tons annually.

Letters to the Editor

The Editor welcomes expression of opinion and fact from responsible persons for publication in these columns. Signed letters are, of course, preferred, but where a desire for anonymity is indicated this will invariably be respected. From time to time letters containing useful ideas and suggestions have been received, signed with a nom-de-plume and giving no information as to their origin. Correspondence cannot be published in THE CHEMICAL AGE unless its authorship is revealed to the Editor.

Professional Unity in the Chemical Industry

To the Editor of THE CHEMICAL AGE.

SIR,—May I enter the lists in acceptance of your invitation and generally in support of your Johnsonian scheme? The times demand new measures and new men. Unless chemists are organised, their work can be to little purpose. They will be unable to make their proper contribution to the restoration of economic order in our social system, of which they should be regarded as a necessary part, a very important part. Academic chemists are so shut up in their test tubes that it is unlikely that of their own accord they will act outside these, in any rational way. They have not enough imagination even to accept the help that is offered. Apparently, they must either be compelled or left to wander in the superior wilderness of their own conceit.

Years ago, I advocated a general union, on the lines adopted by medical men. The explanation of their success is that they had a leader in Dr. MacAlister, who gave himself up to the work. Chemists have no leader, except in America. It is strange that a country which is otherwise leaderless should have a leader in chemistry. I am surprised that Dr. Parsons has not been nominated for presidential office; perhaps it is because he has shown himself to be a leader and Americans have no collective desire to be organised any more than our chemists have. Academic chemists are overcome by their estimate of the value of their labours and are under the absurd impression that they are higher beings who should walk alone. If they continue the habit, they will be simply shelved, of no particular repute in society, though they should be leaders.

The condition of affairs is more than serious. The Chemical Society is moribund, forced to sell out stock to pay for the advertisement of work which most know to be in large part not worth the ink and paper used in writing the record. Mathematical students are not allowed to publish all the trial essays and examples they work out in learning their trade—why should chemical students be advertised as they are? The theories behind most of the work grandiloquently called research are just bunkum. Of course, man has always been a pretender, but the public is beginning to realise that it is not worth while to pay for the pretence of a spurious science. It is becoming more and more a judge of quality. At the moment there is great waste of public funds upon research.

The Society of Chemical Industry is in a better position, because it has attached to its service a considerable number of men of the world, in touch with practice, who count for something socially. It lives on results, not on fancies. None the less, this Society, like all societies, is bound to lose in numbers so long as prosperity remains in abeyance, as it probably will for a long time to come. The one society marked out for real advance is the Bio-chemical Society, as the future of the nations will depend, in the first instance, upon our learning how to feed ourselves properly and economically. This, however, has to raise its social standard, particularly to get rid of a lot of petticoat-stuff and develop a greater sense of accuracy. It must send less unripe fruit to market.

The Chemical Society has a great asset in its library; otherwise its attractions appeal only to very few. The one body that has prospered and must continue to prosper is the Institute of Chemistry. This has been most ably administered and has felt the pulse of the times. It has done what all the other bodies have failed to do! It has held out the idea of chemistry being made a profession and recognised as such—on a par with other liberal professions. Speaking to the Institute recently, I said: "This Institute was founded largely to promote the efficiency of the public analyst—to prevent a very low grade of medical officer taking on the work. It was not wanted for any other purpose. The Chemical Society could have met all needs."

I say this with full knowledge, as I was secretary of the Chemical Society when the Institute unfortunately swarmed off, mainly at the instigation of the late Professor Odling. Not the least of his reasons, I believe, was my advocacy of an enlargement of the scope of the Society, a course which Frankland was prepared to adopt. The game of cross purposes was already in play even in those far off days. In any case, the Chemical Society cannot recover the opportunity it sacrificed, but it can still play a predominant part.

To hold their own, chemists now need to be organised on professional lines into a united body. The Institute is clearly foreshadowed as the central feature; the Chemical Society should give the scientific backbone; the Society of Chemical Industry, the industrial. I would enlarge the title to Institutes of Chemistry and include all the interests of pure and applied chemical science in the one organisation. The Fellows of the Institute are a large majority; let them call upon their organisation to take the lead in forcing on the now essential union of interests.

What rot has set in that there is no man among us coming forward to grasp opportunity? Of what service to us is our science, if we cannot ourselves learn to use it in checking acidity and sweetening dispositions—to the point of learning to live and work together? The one study we need to undertake to-day is into our own efficiency—if we cannot raise this, we shall deservedly sink into a secondary position.—Yours faithfully,

HENRY E. ARMSTRONG.

55 Granville Park,
Lewisham, S.E.13.

The Surgical and Allied Trades

Annual Meeting of the Association

MR. MARK C. RIMMER presided at the fourth annual meeting of the Surgical and Allied Trades Association, held in London on June 14. In his annual report, he said the services rendered to members individually continued to give entire satisfaction. There were two sides to the Association's work. One was assistance to individual firms, chiefly in regard to various trade restrictions and difficulties and the other was the voicing of the collective opinion of the trade and the increasing endeavour to protect the general interest of members. A large number of inquiries had been dealt with. Circulars had been issued giving useful information on matters relating to import duties and restrictions and marking orders. All documents and papers were treated by the secretary as confidential.

Matters connected with the Import Duties Act had received the principle attention of the committee, who lost no time in making representations to the Import Duties Advisory Committee. Following these representations certain medical and scientific instruments and apparatus were specifically exempted from additional duty. Almost continual negotiations had been carried on with H.M. Customs concerning their interpretation of various sections of the Additional Import Duties (No. 1) Order, and although the committee and the Customs authorities did not always see eye to eye on these matters, much good work had been done. But for the existence of the Association their position as traders would long ago have become impossible. There was no doubt they would have to contend with many more attempts at trade restriction. The Import Duties Advisory Committee was receiving representations for additional duties and applications for additions to the Free List. These continual changes were a hindrance to the proper conduct of business and they were fortunate in having such an organisation to look after the interests of the trade and to furnish information on the many complicated points that arose with the operation of any import duties legislation. There had been no further application to the Board of Trade for a Merchandise Marks Order affecting their trade.

The following officers were re-elected:—Chairman, Mr. Mark C. Rimmer; vice-chairman, Mr. Vivian L. Hatrick; hon. treasurer, Mr. H. A. Berens; hon. auditor, Mr. J. Mortimer; executive council, Mr. Chas. Bolter, Mr. H. W. Bush and Mr. A. Searle.

News from the Allied Industries

Artificial Silk

WORKERS in the artificial silk industry in the United States have been given a prolonged holiday throughout the month of June. The most important producer, the American Viscose Company, has closed down all its four plants for that period, and its example has been followed by the Delaware Rayon Company. Contraction of business and accumulation of stocks are responsible for this step.

Safety Glass

A RESOLUTION TO REDUCE THE SHARE CAPITAL from £180,000 to £41,506 11s. by cancelling 1s. 6d. per issued fully-paid share of 2s. will be submitted at an extraordinary meeting of shareholders of Lancegaye Safety Glass, Ltd., convened for July 8. On court sanction, the capital is to be increased to its former amount by the creation of 2,760,869 new ordinary shares of 1s. each. Of these new shares, 200,000 will be offered to shareholders at a discount of 3d. per share. The accounts for 1931 show a loss of £4,896, against £14,254 in 1930.

Soap Trade

ACCORDING TO INFORMATION FROM NEW YORK, Lever Bros. state that the 10 per cent. tax on toilet soaps, which came into effect this week will not be passed on to consumers. Procter and Gamble, and Colgate, Lever's two biggest U.S. competitors, will add the tax to the price of manufactures, thereby intensifying the soap price war.

FORTY-FIVE EMPLOYEES at the Renfrew soapworks of Ogson and Tennant have been presented with gold watches on completion of 15 years' service. Mr. W. W. Watt, managing director of the firm, made the presentations.

Match Industry

IT IS REPORTED FROM NEW YORK that a test case has been brought by a purchaser of $5,000 International Match Bonds against Lee, Higginson and Co., seeking to recover the purchase price of the bonds on the ground that she depended upon material representations of fact which have turned out to be erroneous. The complainant says that the amendment adopted in 1924 giving powers of management to the president of the company was not disclosed to purchasers of bonds, so that they were led to believe that the company enjoyed American management when this was not the case.

Tanning Industry

INQUIRIES ARE BEING MADE in the Midlands for suitable premises for the manufacture of full-chrome leathers, and also for dressing works. Foreign representatives have been inspecting various properties which have been offered for the purpose, and it is expected that a decision will be arrived at shortly. Already some foreign leather manufacturers are operating in the Midlands, in conjunction with one of the present establishments, and a large volume of business has been secured for this new venture. Although there have been previous rumours of inquiries these are the only ones which have so far proved to have any foundation. There is great scope for the manufacture of box and willow calf leathers for shoe uppers, as very few English manufacturers are able to work on the necessary scale.

Artificial Silk

DR. M. FELDMANN, the chairman of Atlas Artificial Silk Processes, Ltd., in a circular issued to shareholders, states that the company is in a position to open up the works and manufacture at once 30,000 lb. of rayon per week if the directors are able to obtain £65,000 working capital. Shareholders are therefore asked to subscribe this amount by taking up 7 per cent. convertible debenture stock. The stock is covered by assets approximately three times, the cost price of the assets being £425,000, and the 7 per cent. interest by the estimated profits four times over. The debenture stock is convertible into ordinary shares at par at any time before June 30, 1935. If every shareholder applies for £40 debenture stock the necessary capital to commence operations will be immediately available. The terms of subscription will be: 25 per cent. on application, 25 per cent. on allotment, 25 per cent. on August 1, and 25 per cent. on October 1.

Non-Ferrous Metals

THE TOTAL STOCK OF REFINED HOME COPPER has been reduced to 18,604 tons by the withdrawal last week of 866 tons, chiefly by the Birmingham district. It is considered probable that there will be further buying, as it is certain that consumers are poorly supplied. On the Metal Exchange activity was experienced in refined metal, about 800 tons having been disposed of by the Copper Exporters' Inc., mostly for early shipment to the Continent. At the reduced sterling rate, based on a 5½c. c.i.f. price, the figure paid for this copper worked out at nearly £34, which is considerably higher than the figure asked by outside sellers in the open market.

China Clay

FORTY-NINE DELEGATES representing the employees in the china clay industry met in conference at St. Austell on June 18, and were addressed by Mr. Ernest Bevin, general secretary of the Transport Workers Union, and Mr. A. Dalgleish, assistant secretary at headquarters. In a statement given to the press it was intimated that a reorganisation scheme designed to secure ninety per cent. of the china clay workers for the Union within the next three months had been endorsed by the conference. Addressing a meeting in the evening, Mr. Matt Giles said their object, getting ninety per cent. of the workers in the industry inside the Union in three months, was not an impossible task. Mr. Dalgleish referred to recent conversations with china clay employers in regard to wages and conditions, and said he wanted the Union to reassert itself in the growing strength of the workers in the china clay field.

Iron and Steel

THE IMPORT DUTIES ADVISORY COMMITTEE has invited Sir W. Charles Wright, chairman of Baldwins, Ltd., to join the National Committee of Iron and Steel Producers, and he has expressed his willingness to serve. The four Regional Committees appointed by the National Committee have elected chairmen as follows:—North East Coast: Mr. E. J. George (Consett Iron Co., Ltd.); Scotland: Mr. John Craig (Colvilles, Ltd.); Midlands: Capt. R. S. Hilton (United Steel Companies, Ltd.); South Wales: Sir John Field Beale, British (Guest, Keen, Baldwins) Iron and Steel Co., Ltd. On Friday, June 17, a meeting was held of all the members of the Special Industries Committees nominated by the Import Duties Advisory Committee under the chairmanship of Mr. C. Mitchell, chairman of the National Iron and Steel Producers' Committee, and these committees elected their chairmen as follows:—Wrought Iron Industry: Mr. C. E. Lloyd (N. Hingley and Sons, Ltd.); Special Steel Industry: Mr. C. W. Kayser (Kayser, Ellison and Co., Ltd.); Armament and Heavy Forging Industries: Mr. A. J. Grant (Thomas Firth and John Brown, Ltd.); Steel Casting Industry: Mr. H. G. Stobart (General Steel Castings Association).

Pottery Manufacture

THE POTTERY (SILICOSIS) REGULATIONS, 1932, which come into operation on July 1, will apply to all factories and workshops in which the manufacture or decoration of pottery, or any process incidental thereto, is carried on, including all factories and workshops in which flint is ground for use in the manufacture of pottery. They provide for periodic medical examination of employees, clearing the floors of potters' shops and the floors of china biscuit placing shops. For the purpose of the regulations, pottery means the manufacture of china and earthenware, including sanitary earthenware, electrical earthenware and earthenware tiles. The provisions of the earlier pottery regulations, in regard to overalls and hand coverings, are to apply to flint milling and all processes involving exposure to powdered flint. Efficient exhaust draughts must be provided in connection with nearly all preliminary pottery processes, and all practicable means must be taken to prevent dust arising. The floors of potters' shops must be rendered impervious and shelves must be provided for moulds. Regarding employees, regulations for overalls, food, ventilation, washing, cleaning of work places and avoidance of dust shall apply to persons employed in the processes of (a) the crushing, grinding, sieving or manipulation of calcined flint preparatory to flint milling; (b) all processes involving exposure to powdered flint.

Chemical Industry Lawn Tennis Tournament

Second Round Results: Third Round Draw

WE have pleasure in announcing that Sir Ernest and Lady Benn have kindly arranged for the final of the Chemical Industry Lawn Tennis Tournament for THE CHEMICAL AGE silver cup to be played at Blunt House, Oxted, early in September. The second round of the second annual tournament was completed on Monday, and the draw for the third round was made on Tuesday. Much more favourable weather prevailed during the period allocated for the second round matches, and some excellent games were played. Of the sixteen fixtures in the second round, fourteen have been played and one scratched. One match remains to be played, namely:—

S. J. Sharman and **H. R. Whitaker**
Williams (Hounslow), Ltd. (Phone: Hounslow 2929.)
v
J. W. Urban and **F. S. Mortimer**
Monsanto Chemical Works, Ltd., (Victoria 1535.)

the result of which will be announced next week. The results of the other matches are given below.

Full details of the third round draw are appended. Competitors should note that all the matches in the third round must be played off by **July 11,** and the results, signed by all four players (winners and losers) must be sent by the winners to the Editor of THE CHEMICAL AGE, Bouverie House, Fleet Street, London, E.C.4, immediately after each match and must reach the office of THE CHEMICAL AGE not later than 9.30 a.m. on July 13. It would facilitate the arrangements of the later rounds if the results could be received as much earlier as possible. Competitors are reminded of the following extracts from the rules of the tournament: (Rule 4) The best of three advantage sets shall be played in all matches. (Rule 8) Except in the case of the final, players drawn against each other must make their own arrangements for playing off their match on a court mutually agreed upon; in the event of disagreement the first pair drawn (as shown in the left hand side of each column) shall have the right to choose the ground (Rule 11). If any players be not present at the agreed place or time of the match, opponents shall be entitled to a walk-over, after having allowed reasonable time (say, a maximum of one hour) for the others appearance. If players find it impossible to play their match on the day originally chosen, they must play it on any other day, to which both sides agree, within the stipulated period.

Second Round Results

J. E. Shirreff and G. G. Stanford (Johnson, Matthey and Co., Ltd.) beat W. G. Baldock and H. W. Drew (Williams, Hounslow, Ltd.) 5—7, 6—3, 6—3.

F. C. White and A. W. White (Howards and Sons, Ltd.) beat F. G. Pretlove and D. B. Hodgson (George Scott and Son, London, Ltd.), 6—3, 10—8.

C. H. B. Jones and Eric Cox (Anglo-Persian Oil Co., Ltd.) beat W. A. Robbins and H. Nichols (Le Grand Sutcliff and Gell, Ltd.), 6—0, 6—1.

E. Thomsett and R. Welsh (British Oxygen Co., Ltd., beat C. Minkleston and W. J. Wood (B. Laporte, Ltd.), 6—4, 6—3.

G. F. Hammond and L. Giltrow (Williams, Hounslow, Ltd.) beat P. Smith and B. T. Francis (Bakelite, Ltd.), 6—0, 6—0.

L. Seabrook and R. Frost (British Oxygen Co., Ltd.) beat F. Hannon and W. Ward (Dussek Bros., and Co., Ltd.), 6—1, 6—0.

A. G. R. Clarke and F. E. Peake (G. A. Harvey and Co., London, Ltd.) beat D. Newton and C. E. Davis (Allen and Hanburys, Ltd., 4—6, 6—3, 6—3.

W. L. Alldis and S. Perridge (Chemicals and Coke Ovens, Ltd.) beat A. A. Killick and G. A. Brittain (B. Laporte, Ltd.), 6—3, 6—2.

S. J. Sharman and H. R. Whitaker (Williams, Hounslow, Ltd.) *v.* J. W. Urban and F. S. Mortimer (Monsanto Chemical Works, Ltd.) —Result to be announced next week.

L. F. Grape and R. C. Mugridge (Borax Consolidated, Ltd.) beat F. Bateman and G. Smith (Dussek Bros. and Co., Ltd.), 6—3, 6—2.

M. B. King and R. H. Hornsby (Howards and Sons, Ltd.) beat A. Baxter (United Yeast Co.) and V. J. Prosser (John Haig and Co.), 6—4, 3—6, 6—3.

A. Collins and H. Sibley (British Oxygen Co., Ltd.) beat C. S. Carter and S. A. Horstman (B. Laporte, Ltd.), 6—1, 6—0.

Idris Williams and T. Clarke (Monsanto Chemical Works, Ltd., Ruabon) walk over, J. S. Boyd (Scottish Dyes, Ltd.) and G. Rae (I.C.I. Nobels, Ltd.) scratched.

C. G. Copp and W. W. Marchant (Doulton and Co., Ltd.) beat W. Collard and E. Thomas (Dussek Bros. and Co., Ltd.), 10—8, 6—1.

S. E. Chaloner and W. Speakman (Monsanto Chemical Works, Ltd., Ruabon) beat A. J. Buck and A. C. Hitchon (Bakelite, Ltd. Birmingham), 6—0, 6—2.

W. B. Miller and G. Lord (British Celanese, Ltd., Spondon) beat E. G. Almond and G. E. Mountney (Bakelite, Ltd., Birmingham), 6—2, 2—6, 6—4.

Third Round Draw

Following are the details of the third round draw. All matches must be played by **July 11,** and the results must reach the Editor of THE CHEMICAL AGE by 9.30 a.m. on July 13 at the latest.

S. E. Chaloner and **W. Speakman**
Monsanto Chemical Works, Ltd., Ruabon, N. Wales. (Phone: Ruabon 3.)
v
C. H. B. Jones and **Eric Cox**
Anglo-Persian Oil Co., Ltd., Britannic House, Finsbury Circus, E.C.2. (Phone: National 1212.)

L. F. Grape and **R. C. Mugridge**
All communications: R. C. Vandermeulen, Borax Consolidated, Ltd., 16, Eastcheap, E.C.3. (Phone: Royal 1450.)
v
A. Collins and **H. Sibley**
British Oxygen Co., Ltd., Angel Road, Upper Edmonton, N.18. (Phone: Tottenham 2488.)

W. B. Miller and **G. Lord**
British Celanese, Ltd., Spondon, Nr. Derby. (Phone: Derby 2200.)
v
Idris Williams and **T. Clarke**
Monsanto Chemical Works, Ltd., Ruabon, N. Wales. (Phone: Ruabon 3.)

C. G. Copp and **W. W. Marchant**
Doulton & Co., Ltd., High Street, Lambeth, S.E.1. (Phone: Reliance 1241.)
v
M. B. King and **R. H. Hornsby**
Howards & Sons, Ltd., Uphall Works, Ilford, Essex. (Phone: Ilford 1113.)

J. E. Shirreff and **G. G. Stanford**
Johnson, Matthey & Co., Ltd., 78, Hatton Garden, E.C.1. (Phone: Holborn 6989.)
v
S. J. Sharman and **H. R. Whitaker** Williams (Hounslow), Ltd. (Phone: Hounslow 2929.)
or
J. W. Urban and **F. S. Mortimer** Monsanto Chemical Works, Ltd., Victoria Station House, Victoria Street, S.W.1. (Phone: Victoria 1535.)

A. G. R. Clarke and **F. E. Peake**
G. A. Harvey & Co. (London), Ltd., Woolwich Road, S.E.7. (Phone: Greenwich 0020, extension 311.)
v
F. C. White and **A. W. White**
Howards & Sons, Ltd., Uphall Works, Ilford, Essex. (Phone: Ilford 1113.)

E. Thomsett and **R. Welsh**
British Oxygen Co., Ltd., Angel Road, Upper Edmonton, N.18. (Phone: Tottenham 2488.)
v
L. Seabrook and **R. Frost**
British Oxygen Co., Ltd., Angel Road, Upper Edmonton, N.18. (Phone: Tottenham 2488.)

G. F. Hammond and **L. Giltrow**
Williams (Hounslow), Ltd. (Phone: Hounslow 2929.)
v
W. L. Alldis and **S. Perridge**
Chemicals & Coke Ovens, Ltd., Vintry House, Queen Street Place, E.C.4. (Phone: Central 1411-2.)

Action against British Celanese, Ltd.

Research Chemists' Claims

IN the King's Bench Division on Wednesday, Mr. Justice Macnaghten had before him an action by Mr. Stanley Joseph Green, of Burton Avenue, Withington, Birmingham, against British Celanese, Ltd., for damages for breach of a contract of employment.

Mr. ROLAND OLIVER, K.C., for the plaintiff, said the contract was a service contract for three years, and then a further three years, and his case was that it was improperly terminated by the defendants.

Plaintiff was a research chemist of considerable distinction. He held first class honors at Cambridge and had held many high positions of great responsibility in the sphere of research chemistry. He was originally employed by the defendants, in 1924. He did a great deal of research work in connection with the production of acetic acid. Plaintiff made some discoveries and was ultimately giving agreement upon which he now sued. The agreement was dated February, 1928, and the service was from January 1, 1928, as a chemist at a commencing salary of £700, rising to £850 for the third year. The agreement, however, ran for four years. The agreement contained a clause that he was not to enter other employ after that period without the defendants' consent, and if they refused their consent he was to receive half his salary.

The contention of the plaintiff was that the action of the defendants amounted in law to a complete repudiation of the whole contract. With regard to the clause that dealt with the defendants' "secrets," the plaintiff was quite willing to give an undertaking not to disclose any confidential information. Plaintiff continued with the defendants till after January, 1931. The defendants had the right to cancel the agreement by six months' notice on certain dates, but failing such notice the agreement would run for another two years. That notice was not given, and the agreement was due to run till the end of 1933. What happened was that in March, 1931, they gave the plaintiff six months' notice. This was contrary to the agreement which provided that notice should be given in January, 1931. The plaintiff's employ, under the circumstances, continued for three years till Janaury, 1934. The notice given, said counsel, was not a valid notice under the agreement and his client was entitled to damages. His client had failed to get other employment.

Plaintiff was called and gave evidence, and said he left defendants' employ in March, 1931, and had been paid his salary for six months. All his efforts to get suitable employment had failed.

Mr. MCCASKIE, K.C., cross-examined for the defendants. Plaintiff said he had applied for all jobs that were suitable which had been advertised between April, 1931, and April of this year. Some of the posts were at £1,000 a year.

Mr. MCCASKIE argued that the notice was not invalid. He invited his lordship to take the view that the agreement provided for six months' notice before the first of July, 1933.

Repudiation of Contract

His lordship, after legal argument, said he had no doubt whatever that the agreement was for a second three years and he admired the ingenuity which could see any other meaning to it. When the defendants' gave the plaintiff notice, he took the only course he could take and that was to contest the matter and seek employment elsewhere to mitigate the damages. Defendants' contention could not be sustained, and plaintiff was entitled to damages for repudiation of the contract. He awarded the plaintiff £1,450 with costs. He further declared that the plaintiff was no longer bound by the restrictive covenant, but he had rightly given an undertaking.

Leave to appeal was given to the defendants on the payment of £500 to the plaintiff.

There was a second action against the same defendants. by Hermon Marden, of Tingley Common, Morley, Leeds, whose salary was £400 a year.

Plaintiff gave evidence as to the efforts he had made to obtain employment.

His LORDSHIP awarded the plaintiff £670 damages with costs, with the same declaration.

Stay of execution was granted on payment of £250 to the plaintiff.

A Wider Use for Coal

Industry, Research and Propaganda

SPEAKING on the wider uses of coal at a luncheon of the Coal Industry Society in London on June 13, Mr. Isaac Foot, Minister of Mines, said that owing to the improvements which had been effected in methods for producing gas and electricity considerably less coal was now required for the production of a given quantity of gas or electricity. Three years ago the National Fuel and Power Committee published a report bringing the information up to date and it was found that in comparing 1930 with 1913 there had been in regard to gas an increase of 45 per cent. in the quantity of gas produced while the increase in the quantity of coal used in the making of gas was only 8½ per cent. As regards electricity, between 1921 and 1930 there had been an increase of 139 per cent. in the number of units generated at electricity stations, whilst the amount of coal used had increased by only 35½ per cent. Had the amount of coal used for these two purposes shown the same increase as the output of gas and electricity there would have been an additional demand for coal amounting to about 12 million tons.

As recently announced, the Mining Association in collaboration with the various organisations of coal distributors and exporters had decided that steps must be taken by the industry to provide means to pay for research and propaganda relating to the extended and progressive use of coal and its products. All the resources of the Department of Mines were available to assist to this end and to encourage the object in view. The Fuel Research Department was started in 1917 and the importance of the work of this organisation had been recognised to the extent that Parliament in recent years had granted over £80,000 a year for that purpose. The essential problems of the coal industry were industrial and not political and if the coal industry at such a time as the present were to endeavour to carry on both a political and an industrial battle at the same time there could be such a dissipation of forces as would have disastrous results. He, therefore, suggested to the industry that it needed all the forces at its command to deal with this industrial problem and to meet the challenge made to the coal industry by other forms of heat and power. The coal industry was not only a great industry; it was also an indispensable industry in this country.

The Mines Department had recently undertaken an inquiry as to the amount of coal consumed in the form of pulverised fuel and the uses to which this had been put. A questionnaire had been sent out to some hundreds of firms all over the country and some interesting replies had been received. When the information had been obtained it would of course be placed at the disposal of the committee which the coal trade had set up.

Applications for Increased Duties

Advisory Committee Invites Representations

THE Import Duties Advisory Committee gives notive of applications for increased duties from a number of trade organisations, including one from the Fertiliser Manufacturers' Association, in respect of superphosphate and compound fertilisers, and one from the India Rubber Manufacturers' Association, Ltd., in respect of rubber footwear. The committee is also considering an application for the adjustment of the duties on paints and colours by substituting the following for the existing class of goods dutiable under Group X (29) of the Additional Import Duties (No. 1) Order, 1932:—Paints, painters' enamels, lacquers, varnishes and printers' inks; distempers, whether dry or not; pigments (whether dry or ground in oil or other medium) other than synthetic organic dyestuffs, colours and colouring matters, dry earth colours, barytes, and carbon black from natural gas.

The committee also gives notice of an application for the addition of magnesium metal and magnesium alloys to the Free List and an application for the reduction of the duties on wicker baskets. Any representations which interested parties desire to make in regard to these commodities should be addressed in writing to the Secretary, Import Duties Advisory Committee, Caxton House (West Block), Tothill Street, London, S.W.1, not later than July 7.

Inventions in the Chemical Industry

Specifications Accepted and Applications for Patents

THE following information is prepared from the Official Patents Journal. Printed copies of Specifications accepted may be obtained from the Patent Office, 25 Southampton Buildings, London, W.C.2, at 1s. each. The numbers given under "Applications for Patents" are for reference in all correspondence up to the acceptance of the Com plete Specification.

Specifications Accepted with Date of Application

MANUFACTURE OF TITANIUM PIGMENTS. Titanium Pigment Co., Inc. Jan. 14, 1930. 374,420

MANUFACTURE OF POLYMERISED ORGANIC COMPOUNDS. Röhn and Haas Akt.-Ges. Feb. 1, 1930. 374,436.

CALCIUM SULPHATE PLASTER MIXES, AND METHODS OF APPLYING THE SAME. Imperial Chemical Industries, Ltd., and V. Lefebure. Feb. 4, 1931. 374,440.

PLASTER AND CEMENT MIXES, AND METHODS OF APPLYING THE SAME. V. Lefebure and Imperial Chemical Industries, Ltd. Feb. 4, 1931. 374,455.

FUELS FOR INTERNAL COMBUSTION ENGINES. D. T. Jones and Imperial Chemical Industries, Ltd. Feb. 12, 1931. 374,481.

CONVERSION OF UNSATURATED ALIPHATIC HYDROCARBONS INTO HIGHER HYDROCARBONS. Ruhrchemie Akt.-Ges. Mar. 1, 1930. 374,422.

METHOD OF PRODUCING AN ELECTROCHEMICAL REACTION BETWEEN THE GASES OF A GAS MIXTURE BY MEANS OF GLOW DISCHARGES. Metallges Akt.-Ges. Apr. 2, 1930. 374,485.

MAKING IRON OXIDE AND SULPHUR DIOXIDE FROM IRON SULPHIDE ORES. H. Freeman. Mar. 5, 1930. 374,486.

MEANS FOR THE SEPARATION OF DUST FROM AIR OR OTHER GASES BY MOISTENING. C. Humphreys and Amalgamated Anthracite Collieries, Ltd. Mar. 4, 1931 374,442.

RUBBER VULCANISATION, AND THE MANUFACTURE OF ACCELERATORS THEREFOR. Goodyear Tire and Rubber Co. Apr. 8, 1930. 374,459.

PROCESS FOR RENDERING FATTY MATERIALS, AND APPARATUS THEREFOR. Darling and Co. Mar. 17, 1930. 374,438.

MANUFACTURE OF KETONES OF THE ANTHRAQUINONE SERIES. A. Carpmael (*I. G. Farbenindustrie*). Mar. 5, 1931. 374,487.

MANUFACTURE OF POROUS ARTILCES CONSISTING OF SILICA. Albright and Wilson, Ltd., and G. King. Mar. 6, 1931. 374,463.

PURIFICATION OF COMBUSTION GASES. R. Lessing. Mar. 10, 1931. 374,467.

MANUFACTURE OF SOLUBLE COMPOSITIONS AND THEIR USE IN DYEING AND PRINTING. Imperial Chemical Industries, Ltd., M. Mendoza, A. G. Murray, and H. B. Briggs. Mar. 11, 1931. 374,497.

MANUFACTURE OF AZO DYESTUFFS. Imperial Chemical Industries, Ltd., and C. Paine. Mar. 11, 1931. 374,498.

PURIFYING ETHYLENE, AND THE PRODUCTION OF ETHYL ALCOHOL THEREFROM. G. F. Horsley and Imperial Chemical Industries, Ltd. Mar. 12, 1931. 374,510.

MANUFACTURE OF HYDROXYCARBOXYLIC ACID MONO-ARYLIDE AND AZO-DYESTUFFS THEREFROM. W. W. Groves (*I. G. Farbenindustrie*). Mar. 26, 1931. 374,548.

CENTRIFUGAL SEPARATORS. H. W. Fawcett. Mar. 26, 1931. 374,549. (Addition to 355,366.)

MANUFACTURE AND PRODUCTION OF METALS, METAL MIXTURES, OR ALLOYS. J. Y. Johnson (*I. G. Farbenindustrie*). Apr. 2, 1931. 374,500.

CATALYTIC HYDROGENATION OF CARBONACEOUS MATERIALS. Chemical Reactions, Ltd. (*Deutsche Gold und Silber-Scheidean-stalt vorm. Roessler*). Apr. 10, 1931. 374,507.

PROCESS FOR THE MANUFACTURE OF DISULPHIDES FROM MERCAPTO COMPOUNDS. I. G. Farbenindustrie. May 9, 1930. 374,594.

REACTIVATION OF ADSORBENTS. Gas Light and Coke Co., R. H. Griffith, and R. S. Chaplin. May 12, 1931. 374,597.

APPARATUS AND PROCESS FOR THE DISTILLATION OF HIGH-BOILING SUBSTANCES (OILS AND THE LIKE). E. Lutz. July 18. 1930. 374,662.

METHOD OF PREPAKING IMIDAZOLYL-PROPIONIC ACID FROM GLUTAMIC ACID. S. Akabori and S. Numano. July 24, 1931. 374,674.

RECOVERING BERYLLIUM FROM A SILICATE ORE THEREOF. Beryllium Development Corporation. Sept. 17, 1930. 374,705.

FILLING MATERIAL OR PACKING FOR ABSORPTION, WASHING, OR REACTION TOWERS, REFLUX CONDENSERS SUCH AS FRACTIONATING COLUMNS, OR THE LIKE. G. Harrington. Aug. 31, 1931. 374,707.

MANUFACTURE OF PURIFIED ESTERS OF PHOSPHORIC ACID AND THIOPHOSPHERIC ACID. Monsanto Chemical Works. Sept. 29, 1930. 374,721.

Complete Specifications open to Public Inspection

MANUFACTURE OF DISINFECTING, ANTI-PARASITIC AND INSECTICIDAL AGENTS. Soc. Anon. des Mines D'Orbagnoux. June 13, 1931. 7790/32.

MANUFACTURE OF LUBRICATING OILS. Standard Oil Development Co. June 11, 1931. 13214/32.

MANUFACTURE OF PHOSPHORUS. Victor Chemical Works. June 8, 1931. 14722/32.

PROCESS OF OBTAINING ALCOHOLS STARTING FROM ETHYLENIC HYDROCARBONS. Soc. des Carburants Synthetiques. June 11, 1931. 14867/32.

HEAT-TREATING, THERMALLY UNITING, OR THERMALLY DIVIDING METALS. Linde Air Products Co. June 8, 1931. 15051/32.

ADSORPTION OF GASES OR VAPOURS. Silica Gel Corporation. June 8, 1931. 16111/32.

PROCESS AND MEANS FOR THE PRODUCTION OF IONISATION. G. Brion and A. J. Krutzsch. June 13, 1931. 16331/32.

PROCESS FOR THE SEPARATION OF GASEOUS MIXTURES. Soc. L'Air Liquide, Soc. Anon. Pour L'Etude et l'Exploitation des Procedes G. Claude. June 13, 1931. 16334/32.

PROCESS FOR THE MANUFACTURE OF A FIELD WEED-KILLER. Chemische Fabrik L. Meyer. June 10, 1931. 16376/32.

PRODUCTION OF AMMONIUM SULPHATE. Appareils et Evaporateurs Kestner. June 10, 1931. 16405/32.

PROCESSES OF COALESCING METALS. Copper Deoxidation Corporation. June 10, 1931. 16419/32.

PROCESS FOR THE TREATMENT OF THE REACTION MIXTURES FORMED IN THE PREPARATION OF ACETIC ANHYDRIDE BY THE THERMAL DECOMPOSITION OF ACETIC ACID. C. F. Boehringer and Soehne Ges. June 11, 1931. 16586/32.

FIBROUS OR FILAMENTARY MATERIALS, AND PROCESSES FOR TREATING SAME. Naugatuck Chemical Co. June 12, 1931. 16618/32.

ARRANGEMENT FOR THE REMOVING OF SOLID OR LIQUID PARTICLES FROM GASES OR VAPOURS. E. Haber. June 12, 1931. 16669/32.

MANUFACTURE OF BISMUTH SALTS OF ARSENO-COMPOUNDS. I. G. Farbenindustrie. June 11, 1931. 16683/32.

PROCESS FOR THE ESTERIFICATION OF ORGANIC ACIDS WITH SEPARATION OF THE ESTERS IN HIGHLY CONCENTRATED FORM. Invention Ges für Verwaltung und Verwertung Chemisch-Technischer Patente Ges. June 13, 1931. 16710/32.

PROCESS FOR MANUFACTURING HYDROPHILOUS FATTY SUBSTANCES. Deutsche Dydrierwerke A.-G. June 12, 1931. 16725/32.

Amended Specification Published

EMULSIONS OF RESINS, ETC. Imperial Chemical Industries, Ltd., and another. 349,988.

Applications for Patents

MANUFACTURE OF ANHYDROUS BORAX. American Potash and Chemical Corporation and F. B. Dehn. June 14. 16822.

MANUFACTURE, ETC., OF VAT DYESTUFFS. I. B. Anderson, Imperial Chemical Industries, Ltd., D. C. R. Jones, and R. F. Thomson. June 13. 16727.

TREATMENT OF SULPHIDE LEAD CONCENTRATES, ETC. R. Bingham, Huntington, Heberlein and Co., Ltd., and A. Pearson. June 17. 17194.

CATALYTIC REACTIONS. G. Botella Peréz. June 14. (Spain, May 10.) 16810.

DISTILLATION TREATMENT OF MATERIALS CONTAINING HYDROCARBONS E. W. Brocklebank and W. B. Mitford. June 14. 16849.

MANUFACTURE OF MONOAZO DYESTUFFS. A. Carpmael (*I. G. Farbenindustrie*). June 14. 16832.

PRODUCTION OF ALCOHOL FREE FROM FUEL OIL. Deutsche Gold-und Silber Scheideanstalt vorm. Roessler. June 18. (Germany, June 19, '31.) 17312.

MANUFACTURE OF HYDROPHILOUS FATTY SUBSTANCES. Deutsche Hydrierwerke Akt.-Ges. June 13. (Germany, June 12, '31.) 16725.

MANUFACTURE OF COMPOSITIONS CONTAINING SOLID-GELLED OILS. E. I. Du Pont de Nemours & Co. June 18. (United States). June 20, '31.) 17328.

CATALYTIC REDUCTION OF ORGANIC COMPOUNDS. E. W. Fawcett and Imperial Chemical Industries, Ltd. June 17. 17242.

THERMAL TREATMENT OF HYDROCARBONS. J. Y. Johnson (*I. G. Farbenindustrie*). June 18. 17284.

MANUFACTURE OF BISMUTH SALTS OF ARSENO-COMPOUNDS. I. G. Farbenindustrie. June 13. (Germany, June 11, '31.) 16683.

PRODUCTION OF ANTIMONATES OF ALKALI, ETC., EARTH METALS. I. G. Farbenindustrie. June 15. (Germany), July 3, '31.) 16903.

MANUFACTURE OF CHLORO- AND BROMO-DERIVATIVES OF 2-AMINONAPHTHALENE-1-SULPHONIC ACID. I. G. Farbenindustrie. June 15. (Germany, June 15, '31.) 16950.

MANUFACTURE OF PURE TITANIUM DIOXIDE. I. G. Farbenindustrie. June 15. (Germany, June 15, '31.) 16965.

MANUFACTURE OF NEW POLYAZO DYESTUFFS. I. G. Farbenindustrie. June 17. (Germany, June 17, '31.) 17219.

MANUFACTURE OF MOULDED POLYMERISATION PRODUCTS. I. G. Farbenindustrie. June 18. (Sept. 29, '31.) (Germany, Oct. 2, '30.) 17285.

CARBONISATION OF COAL, ETC. Illingworth Carbonisation Co., Ltd. and S. R. Illingworth. June 14. 16831.

PRODUCTION OF MOTOR FUELS. Imperial Chemical Industries, Ltd. June 13. 16728.

Weekly Prices of British Chemical Products

Review of Current Market Conditions

The following notes on the chemical market conditions in Great Britain are based on direct information supplied by the British manufacturers concerned, and unless otherwise qualified the figures quoted apply to fair quantities, net and naked at makers' works. Where no locality is indicated, the prices are general for the United Kingdom. Particulars of the London chemical market are specially supplied to THE CHEMICAL AGE by R. W. Greeff and Co., Ltd., and Chas. Page and Co., Ltd., and those of the Scottish chemical market by Chas. Tennant and Co., Ltd.

IN the London market, prices have been fairly steady, with very little change. The demand for chemicals has been a little better. Prices for coal tar products remain firm, and the market is unchanged from last week. Although the price tendency of the vast majority of products on the Manchester chemical market is undoubtedly steady, it has again to be reported that the few changes of consequence that have occurred during the past ewek have been slight recessions. With regard to current business, the uncertain labour situation in the Lancashire cotton textile industry militates against forward contract buying, and although fairly satisfactory deliveries are reported against existing contracts, most of the new bookings relate to early delivery positions. Home business has been slightly better during the week in the Scottish market. Export business remains quiet.

General Chemicals

ACETONE.—LONDON: £65 to £68 per ton; SCOTLAND: £66 to £68 ex wharf, according to quantity.

ACID, ACETIC.—Tech. 80%, £37 5s. to £39 5s.; pure 80% £38 5s. to £40 5s.; tech., 40%, £19 15s. to £21 15s.; tech., 60%, £28 10s. to £30 10s. SCOTLAND: Glacial 98/100%, £48 to £50; pure 80%, £38 5s.; tech. 80%, £37 5s. d/d buyers' premises Great Britain. MANCHESTER: 80%, commercial, £39; tech. glacial, £52.

ACID, BORIC.—SCOTLAND: Granulated commercial, £26 10s. per ton; B.P. crystals, £35 10s.; B.P. powder, £36 10s. in 1-cwt. bags d/d free Great Britain in one-ton lots upwards.

ACID, CHROMIC.—11d. per lb., less 2½% d/d U.K.

ACID, CITRIC.—1s. 1d. per lb. LONDON: 1s. 1½d., less 5%. MANCHESTER: 1s. 0¾d.

ACID, CRESYLIC.—97/99%, 1s. 6d. to 1s. 8d. per gal.; 99/100%, 1s. 9d. to 2s.

ACID FORMIC.—LONDON: £50 per ton. *£52.

ACID, HYDROCHLORIC.—Spot, 3s. 9d. to 6s. carboy d/d according to purity, strength and locality. SCOTLAND: Arsenical quality, 4s.; dearsenicated, 5s. ex works, full wagon loads.

ACID, LACTIC.—LANCASHIRE: Dark tech.. 50% by vol., £23 10s. per ton; 50% by weight, £27 10s.; pale tech., 50% by vol., £27; 50% by weight, £32; 80% by weight, £52; edible, 50% by vol., £40. One-ton lots ex works, barrels free.

ACID, NITRIC.—80° Tw. spot, £20 to £25 per ton makers' works, according to district and quality. SCOTLAND: 80°, £23 ex station full truck loads.

ACID, OXALIC.—LONDON: £45 10s. per ton in casks, £48 10s. to £52 10s. in kegs. SCOTLAND: 98/100%, £49 to £52 ex store. MANCHESTER: £47, ex store.

ACID, SULPHURIC.—Average prices f.o.r. British makers' works, with slight variations owing to local considerations: 140° Tw. crude acid, £3 per ton; 168° Tw. arsenical £5 10s.; 168° Tw. non-arsenical, £6 15s. SCOTLAND: 144° quality, £3 12s. 6d.; 168°, £7; dearsenicated, 20s. per ton extra.

ACID, TARTARIC.—1s. 0¾d. per lb. SCOTLAND: B.P. crystals, 1s. 1d. to 1s. 1¼d., less 5% carriage paid. MANCHESTER: 1s. 0¾d.

ALUM.—SCOTLAND: Lump potash, £9 per ton ex store.

ALUMINA SULPHATE.—LONDON: £8 5s. to £9 10s. per ton. *£7 10s. SCOTLAND: £8 to £8 10s. ex store.

AMMONIA, ANHYDROUS.—Spot, 10d. per lb. d/d in cylinders. SCOTLAND: 10d. to 1s. containers extra and returnable.

AMMONIA, LIQUID.—SCOTLAND: 80°, 2¾d. to 3d. per lb. d/d, according to quantity.

AMMONIUM CARBONATE.—SCOTLAND: Lump, £36 per ton; powdered, £38, in 5-cwt. casks d/d U.K. stations or f.o.b. U.K. ports.

AMMONIUM CHLORIDE.—£37 to £45 per ton, carriage paid. LONDON: Fine white crystals, £19 to £20. (See also Salammoniac.)

AMMONIUM CHLORIDE (MURIATE).—SCOTLAND: British dog tooth crystals, £32 to £35 per ton, carriage paid according to quantity. (See also Salammoniac.)

AMMONIUM BICHROMATE.—8d. per lb. d/d U.K.

ANTIMONY OXIDE.—SCOTLAND: Spot, £24 per ton, c.i.f. U.K. ports.

ANTIMONY SULPHIDE.—Golden 6½d. to 1s. 1½d. per lb.; crimson, 1s. 4d. to 1s. 6d. per lb. according to quality.

ARSENIC.—LONDON: £24 10s. c.i.f. main U.K. ports for imported material; Cornish nominal, £26 10s. f.o.r. mines. SCOTLAND: White powdered £27 ex wharf; spot, £27 10s. ex store. MANCHESTER: White powdered Cornish, £26 10s. at mines.

ARSENIC SULPHIDE.—Yellow 1s. 6d. to 1s. 8d. per lb.

BARIUM CHLORIDE.—£11 to £11 10s. per ton.

BISULPHIDE OF LIME.—£7 10s. per ton f.o.r. London, packages free.

BLEACHING POWDER.—Spot 35/37% £7 19s. per ton d/d station in casks, special terms for contract. SCOTLAND: £8 15s. in 5/6 cwt. casks.

BORAX, COMMERCIAL.—Granulated £15 10s. per ton, powder £17, packed in 1-cwt. bags, carriage paid any station Great Britain. Prices are for 1-ton lots and upwards.

CADMIUM SULPHIDE.—3s. 6d. to 3s. 9d. per lb.

CALCIUM CHLORIDE.—Solid 70/75% spot £5 5s. to £5 15s. per ton d/d station in drums.

CARBON BISULPHIDE.—£30 to £32 per ton, drums extra.

CARBON BLACK.—5d. to 5⅞d. per lb., ex wharf.

CARBON TETRACHLORIDE.—£45 to £55 per ton, drums extra.

CHROMIUM OXIDE.—10d. to 10½d. per lb. according to quantity d/d U.K. Green 1s. 2d. per lb.

CHROMETAN.—Crystals 3¾d. per lb. Liquor £19 10s. per ton d/d.

COPPERAS (GREEN).—SCOTLAND: £3 15s. per ton, f.o.r., or ex works.

CREAM OF TARTAR.—LONDON: £5 to £5 3s. 6d. per cwt.

FORMALDEHYDE.—LONDON: £27 to £27 10s. per ton. *£30. SCOTLAND: 40%, £28 10s. ex store.

HYDROGEN PEROXIDE.—LONDON: *100 vols. 10d. per lb.

LAMPBLACK.—£46 to £50 per ton.

LEAD, ACETATE.—LONDON: White, £39 to £41 per ton. Brown £1 per ton less. SCOTLAND: White Crystals £42 to £44 c.i.f. U.K. ports. Brown £1 per ton less. MANCHESTER: White, £35; Brown, £33 10s.

LEAD NITRATE.—£28 per ton.

LEAD, RED.—SCOTLAND: £28 10s. per ton d/d buyer's works.

LEAD, WHITE.—SCOTLAND: £40 per ton carriage paid.

LITHOPONE.—30%, £20 to £22 per ton.

MAGNESITE.—SCOTLAND: Ground Calcined £9 per ton ex store.

METHYLATED SPIRIT.—61 O.P. Industrial 1s. 8d. to 2s. 3d. gal. Pyridinised Industrial, 1s. 10d. to 2s. 5d. Mineralised, 2s. 9d. to 3s. 3d. 64 O.P. 1d. extra in all cases. Prices according to quantities. SCOTLAND: Industrial 64 O.P., 1s. 9d. to 2s. 4d.

NICKEL AMMONIUM SULPHATE.—£38 per ton d/d.

NICKEL SULPHATE.—£38 per ton d/d.

POTASH, CAUSTIC.—£30 to £33 per ton. LONDON: £42. MANCHESTER: £40.

POTASSIUM BICHROMATE.—Crystals and Granular, 5d. per lb. net d/d U.K. Discount according to quantity. Ground 5¾d. LONDON: 5d. per lb. with usual discounts for contracts. SCOTLAND: 5d. d/d U.K. or c.i.f. Irish Ports with allowance for contracts. MANCHESTER: 5d.

POTASSIUM CARBONATE.—SCOTLAND: 96/98% spot £28 per ton ex store. LONDON: £31 10s. to £32. MANCHESTER: £31.

POTASSIUM CHLORATE.—3¾d. per lb. export London in 1-cwt. kegs. LONDON: £37 to £40 per ton. SCOTLAND: 99¾/100% powder, £34. MANCHESTER: £36.

POTASSIUM CHROMATE.—6½d. per lb. d/d U.K.

POTASSIUM NITRATE.—SCOTLAND: Refined Granulated £28 per ton c.i.f U.K. ports. Spot £30 per ton ex store.

POTASSIUM PERMANGANATE.—LONDON: 8½d. to 9d. per lb. SCOTLAND: B.P. crystals, 8½d. MANCHESTER: Commercial, 8d.; B.P., 8½d.

* Prices quoted by other manufacturers.

Other Price Changes

PRESSURE on space in this week's issue of THE CHEMICAL AGE makes it necessary to omit the second page of Weekly Prices, in which there are no price changes to report other than the following:—

SULPHATE OF COPPER.—MANCHESTER: £16 per ton f.o.b.

REFINED COAL TAR.—SCOTLAND: 4¾d. to 5d. per gal.

ACETATE OF LIME.—Brown, £8 per ton; grey, £12. Liquor, 4d. to 5d. per gal. MANCHESTER: Brown, £7 5s.; grey, £11.

Nitrogen Fertilisers

SULPHATE OF AMMONIA.—The export market continued quiet and the price was unchanged at £4 5s. per ton f.o.b. U.K. port in single bags. It is not anticipated that there will be much interest in this product until next season's prices are announcede. The small lots now required at home are selling at from £5 10s. to £6 5s. per ton delivered in 6-ton lots to consumers' nearest station. The amount of business transacted is small.

NITRATE OF SODA.—Imported nitrate remains at £9 per ton, and British at £8 16s. delivered in 6-ton lots.

NITRO-CHALK.—The price remains at £7 5s. per ton delivered in 6-ton lots.

From Week to Week

THE CONTINENTAL TINTEX AND DYE PRODUCTS have removed their offices to 4 Queen Victoria Street, London, E.C.4.

F. H. FAULDING AND CO., LTD., have removed from 76 Finsbury Pavement, to Spencer House, South Place, London, E.C.2. Their telephone number and telegraphic address remain unaltered.

THE MEMBERS OF THE MIDLAND SECTION of the Coke Oven Managers' Association visited the plant of the Yorkshire Coking and Chemical Co., Ltd., at Glass Houghton, on June 14. The manager of the works is Mr. T. P. Carr, chairman of the section.

SENOR ENRIQUE ZANARTU, the new Chilean Finance Minister, is reported to have assured the Cosach interests that the settlement to be reached will be equitable and acceptable to all the parties concerned, and will maintain unimpaired the sources of national wealth.

MR. R. SCARGILL, an assistant chemist at the works of L. B. Holliday and Co., Ltd., at Deighton, Huddersfield, was badly burnt on the face and hands by caustic potash at the works on June 18. He was working in the laboratory when a mishap occurred to the heater.

THE ANGLO-CHILEAN CONSOLIDATED NITRATE CORPORATION, an important member of the Cosach combine, has announced that, owing to the present disturbed conditions, payment of the half-yearly interest, due on July 1, on the 7 per cent. First Mortgage Debenture stock, is unavoidably postponed.

UPON HIS RETIREMENT from the directorate and from active participation in the works management of the Monsanto Chemical Works Ltd., at Cefn, North Wales, Dr. H. Kippenberg has been presented by the staff and workpeople with a portable gramophone. As a parting gift, Dr. Kippenberg presented the recreation club with a piano.

THE HONORARY DEGREE of Doctor of Engineering, Sheffield University, has been conferred on Mr. J. H. W. Laverick, managing director of the Tinsley Park Colliery Co., Ltd., Sheffield. Mr. Laverick has been associated with the coal mining industry for 50 years and is chairman of the Mining and Fuel Committee at Sheffield University.

HUNDREDS OF TONS OF WAXED PAPER were destroyed in a fire at a factory on the site of the Palace of Industry in Wembley Exhibition grounds on June 20. The building involved was a store belonging to Griffith and Diggens, and adjoins their factory where waxed paper and other products are manufactured. The flames spread with great rapidity, and an hour and a half after the alarm was given, the flames were still raging so fiercely that additional lengths of hose were hurried to the scene in private motor cars. After three hours' work the fire was got under control. It was almost entirely confined to the store, and the main factory was not damaged except by water.

TWO WOMEN WERE KILLED in an explosion which occurred on June 16 in one of the sheds of the firework factory of Joseph Wells and Sons, at Honor Oak Park, London. The victims were Edith Maynard (23) of Catford, and Winifred Norris (21), of Forest Hill. The explosion occurred in one of the sheds where crackers were being filled and the building immediately burst into flames. The sound of the explosion alarmed the whole neighbourhood, and in the Honor Oak and Forest Hill Golf Club, a short distance from the factory, electric light globes were smashed and ornaments and furniture were flung to the ground. At the inquest on June 21, a suggestion was put forward that the explosion was caused by the heat of the sun on the day in question.

THE IMPORTANCE OF TECHNICAL EDUCATION in the clay industry was stressed by Mr. Leonard Timmins, of Stoke-on-Trent, the newly elected president of the National Association of Clayworks Managers, at the association's banquet at Buxton on June 20. Mr. Timmins said the clay industry had only just begun to avail itself of the technical knowledge that had been obtained for it. Not only the managers but every member of every staff should be technically instructed. Admirable work was being done in the colleges of Sheffield, Leeds, and Stoke-on-Trent, but if everyone in the clay industry demanded instruction the facilities would not be extensive enough to provide it. Laboratories were instituted in many factories, but accompanying the laboratory there should be a classroom where regular instruction could be given to the young people employed at the factory.

RECENT WILLS include :—Mr. William Cock, of 1101 Ashfield Terrace, Slaithwaite, Huddersfield, chairman of George Cock, Ltd., of Longfield Dyeworks, Linthwaite, £18,873, (net personalty £18,789); Mr. Albert Victor Derry, of Tapton Court, Sheffield, coke oven and consulting engineer, for many years managing director of the Koppers Coke Oven Co., Ltd., and a director of the British Tar Products, Ltd., of Irlam, Manchester, £53,314 (net personalty £43,084); The Hon. Cecil Henry Campbell (83), of Bryanston Square, London, son of the third Lord Stratheden and Campbell, a director of the New Transvaal Chemical Co., and the Staveley Trust, £18,989 (net personalty £14,924); Mr. Frederic Lennard, of Colwood Court, Warninglid, Sussex, for many years head of Forbes, Abbott, and Lennard, Ltd., chemical manufacturers, of 52 Grosvenor Gardens, S.W., £163,871 (net personalty £126,461.)

THE LONDON OFFICE of Crofts (Engineers), Ltd., of Thornbury, Bradford, has been removed from 46 Cannon Street, to Oswaldestre House, 34 Norfolk Street, W.C.2. The company's new telephone number is Temple Bar 6077, and the telegraphic address is now "Croftpul, Estrand, London."

THE DEPARTMENT OF AGRICULTURE FOR SCOTLAND announce that in pursuance of the powers conferred on them by Section 23 of the Fertilisers and Feeding Stuffs Act, 1926, they propose to make new regulations to replace the Fertilisers and Feeding Stuffs Regulations, 1928, dated May 23, 1928. It is proposed also to bring the new regulations into operation on September 1, 1932.

DR. CECIL B. MARSON, chief chemist to the Northern Coke Research Committee, has been appointed Head of the Chemistry Department of the Hull Municipal Technical College. Dr. Marson, who is a native of Hull, commenced his career in the department of the East Riding County Analyst at Hull, and after the War spent eight years at the Leeds University, where, in connection with research into fuel problems, he was awarded the degrees of M.Sc. and Ph.D. He has been three years with the Northern Committee at Newcastle.

IN AN AFTER-LUNCHEON SPEECH at the annual conference of the National Association of Clayworks Managers at Buxton, June 20, Mr. W. J. Gardner, vice-president of the Ceramic Society and the British Refractories Research Association, appealed for financial support for the research work which is being carried on by these two organisations. Hitherto, he said, the Government and the clay industry had been contributing equally to the funds of the British Refractories Research Association, but now the association had been given notice that Government support would cease altogether unless more money was forthcoming from the clay industry.

Obituary

THE RT. HON. JOHN WILLIAM WILSON, chairman of Albright and Wilson, Ltd., chemical manufacturers, Oldbury, on Saturday, June 18, aged 73. Mr. Wilson was Liberal member for Parliament from 1895 to 1922. He was born at Edgbaston on October 22, 1858, was educated at Grove House School, Tottenham, and in Germany, and entered business in the chemical manufacturing firm at Oldbury, of which he later became a partner. He was first elected to the House of Commons for Oldbury, as a Liberal Unionist. He broke with Mr. Balfour and his party, however, on the educational and licensing questions, and in the election of 1906 stood and was returned as a Liberal. Later he was member for Worcestershire North.

W. J. Bush and Co. Ltd.

Annual General Meeting

PRESIDING at the 35th annual general meeting of the shareholders of W. J. Bush and Co., Ltd., held in London on June 8, Mr. James M. Bush, chairman and managing director, said it is too early to judge the effect of the Import Duties Act passed in March last. It was obviously an emergency Act, designed both to raise revenue and to reduce the adverse foreign trade balance of the country. Many of the company's raw materials not produced within the Empire bear the 10 per cent. tax, which makes it difficult, and in some cases impossible, to meet foreign competition in export markets. They were also placed at a disadvantage in export distributing trade in many articles. The position has, therefore, been put before the Advisory Committee by the trade associations of which the company is a member.

Mr. G. W. Bishop congratulated the board on the result of their efforts in the past year, and asked if the chairman could make some comment on the present international trade position.

The chairman said he thought that the British Government, by passing the Gold Standard (Amendment) Act and the Import Duties Act, had done the best it could up to the present to meet the situation, and he personally attached the greatest importance to the promise of closer economic union with the Dominions as an outcome of the Ottawa Conference.

The chairman, in proposing the re-election of Dr. P. C. C. Isherwood as a director, said that during the 30 or more years in which that gentleman had been in the service of the company he had proved himself an indefatigable and able worker.

Forthcoming Events

June 28.—The National Physical Laboratory. Annual visit of inspection. 3 to 6.30 p.m. Teddington.

The Dyestuffs Monthly Supplement

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Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.*

A New Delustring Process

A method of delustring cellulose acetate fabrics which, when applied to crepe-back satins with cellulose acetate rayon warps and silk wefts, serves the double purpose of delustring the rayon and degumming the silk, is described by J. Sterling in a recent issue of the *Textile World*. The fabric is first hooked off into 36-in. folds, tied with strings and suspended from sticks arranged so that the loops hang in a tub. The fabric is treated with water at about 100° F. and the temperature is raised gradually to 120° F. The fabric is then laid up at the end of the trough and 8 to 10 per cent. of olive-oil soap (based on the weight of the goods) and 1 oz. soda ash per 30 gallons of liquor are added to the water. The goods are then run back into this soap bath at 120° F., and the temperature is raised gradually to at least 175° F. At the end of the first hour the degumming of the silk should be almost complete. A quick test of this is to take a silk thread and untwist it. If the filaments separate, the degumming is complete. When a satisfactory boil-off has been obtained the temperature of the bath is raised to 185° to 195° F. Treatment for about 30 minutes at this temperature will usually give sufficient delustring. The goods should be removed on the first signs of saponification which is indicated by the appearance of affinity for direct dyes. If a batch shows signs of saponification and the silk is degummed but the lustre still too high, a fresh bath of water alone heated to the required temperature will serve to delustre without risk of further saponification. After the delustring treatment the goods are rinsed and dyed in the usual way.

A New I.C.I. Caledon Colour

Caledon Dark Blue GS, an addition to the range of Caledon dyestuffs produced by Imperial Chemical Industries, Ltd., is suitable for application to all types of cotton goods, either in the open beck or in machines, and is particularly recommended for use in the dyeing of piece goods on account of its excellent levelling properties. It is also of considerable interest for the dyeing of viscose, linen and silk. In common with Caledon Dark Blues 3BS and WFS, this product possesses excellent fastness to moisture. It is dyed according to the process given as Method I in the I.C.I. pattern card "Caledon and Durindone Colours." Vatting and dyeing should be done at 140° F. For printing Caledon Dark Blue GS is suitable for application by the potash-formosul method. Regarding dyeing and fastness properties, it is pointed out that levelling, affinity, hot pressing, cross dyeing, action to light, washing and mercerising qualities are excellent. Resistance to acids, both organic and mineral, is excellent; resistance to alkalies is very good. The only change noticed in artificial light is a slight redding of the shade.

Chloramine Brilliant Orange RS

Brightness of shade and very good solubility are the chief characteristics of this new colour, which is produced by the Sandoz Chemical Co., Ltd. These properties mark a distinct advance on Chloramine Fast Orange S, the relatively poor solubility of which has caused it to have little use in machine dyeing. The new colour is, moreover, less affected by hard water than similar brands. Although its fastness to light may be considered as good, the Brilliant Orange is not equal in this respect to the Chloramine Fast Oranges or to the Solar Oranges, which, however, it surpasses in other ways. The fastness of this colour to chlorine is moderately good, and it can be recommended for the dyeing of self-shades where fastness to heavy washing is not required. The fastness to acids and alkalis is excellent; to rubbing and ironing, good. Chloramine Brilliant Orange RS is particularly suitable for the dyeing of irregular viscose or cuprammonium rayons and of mixed viscose-cotton materials. It is also suitable for dyeing half-wool and half-silk materials, the animal fibres of which are reserved. Very bright shades are produced on unweighted silk when the colour is applied in an acid bath. For weighted silk it has little affinity. The colour is discharged to a white by hydrosulphite. Chloramine Brilliant Orange RS can be used for paper dyeing, and in the preparation of lakes. It is also applicable to chrome leather.

Spring Silk Colours

Seventy-eight shades appear on one of American Spring cards. Blues are highlighted on this new card in bright medium tones with a slight purplish cast, marine and light navy shades reflecting the nautical influence, and deep azure tones with a hint of violet. Significant as a high style note are the new light beiges in the shell and bisque tints. Creamy champagne tones shading into light mocha represent another smart range. Spring browns are interpreted in creamy chocolate shades. Reds are shown in brilliant yellowish or orange hues and bright apple red. New Spring developments of copper and rust are supplemented by subtle peach-apricot tones of coppery cast. Lively shades of yellowish green continue their popularity. The newest repetition of this colour family, however, is a light olive-tinted range. Soft jade tones, like those reflected in Chinese pottery, constitute another favoured version of green. Another new colour note portrayed on the card comprises a range of deep rich pinks shading into cherry rose. Lighter Spring renditions of grey are represented in clear pastel and medium tones.

Stripping Cellulose Acetate Rayon

According to a recent communication to the *Textile Manufacturer*, it is possible to lighten a dyeing on cellulose acetate rayon by treatment with soap at about 80° C., but a complete removal of the colour is almost impossible. On a small scale good results can be obtained by the use of a bath containing 5 c.c. of sodium hypochlorite and 1 c.c. of formic acid per litre, but this mixture cannot be used on a large scale owing to the quantity of chlorine which is evolved. A recently patented method, which gives an almost perfect stripping, consists of treating the dyed material in a bath containing 2 grams of decolorising charcoal and 5 grams of soap per litre, at a temperature of 80° to 85° C., until sufficient colour has been removed. In order to obviate difficulties in the subsequent washing-out of the finely divided charcoal, protective colloids, such as glucose, dextrine, or glue may be added to the bath, but excess should be avoided. This treatment does not alter the tone of the dyeing, so that even if the colour is not stripped down to a perfect white, there is no difficulty in redyeing to the proper shade. When mixed fabrics are

to be stripped, any other decolorising agent may be added to the charcoal mixture to suit the nature of the dyes on the cotton or other fibres present.

The British Colour Council

No fewer than 350 concerns have been enrolled in the British Colour Council since its formation last year, and work of considerable importance has been accomplished. There is still much to be done, however, in the direction of establishing the principle that instead of looking to other countries for guidance as to the colours to be used in the manufacture of textiles and other products, Britain should determine the colours for itself. Apart from conducting research connected with colours, one of the objects of the Council is to co-ordinate and standardise the names and numbers of all colours. It aims at providing its members with the latest and most correct colour information from all sources, obtaining advance information about colour tendencies in fashions, limiting the speculative element in colour decisions and warning members when any of these colours are unexpectedly changed. Various cards are issued showing the colours recommended—40 different colours for woollen fabrics, 60 for silk and silk mixtures, 12 for hosiery, and so on. The results of the co-ordinated efforts of the Council promise to be of great benefit to the whole of the dyestuffs interests.

Dyestuff Licences for December

The following statement relating to applications for licence under the Dyestuffs (Import Regulation) Act, 1920, made during December has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee. The total number of applications received during the month was 434, of which 329 were from merchants or importers. To these should be added 19 cases outstanding on November 30, making a total for the month of 453. These were dealt with as follows :—Granted, 424 (of which 405 were dealt with within seven days of receipt) ; referred to British makers of similar products, 27 (of which 20 were dealt with within seven days of receipt) ; outstanding on December 31, two. Of the total of 453 applications received, 425 or 94 per cent. were dealt with within seven days of receipt.

An Aspect of Union Dyeing

Difficulties Attending the Dyeing of Garments

For his paper read before the Manchester Section of the Society of Dyers and Colourists on Friday, December 11, Mr. A. J. Crockett took the subject of garment dyeing. He said that wool when chlorinated to render it unshrinkable was very difficult to dye satisfactorily. The practice in the dyehouse with which he was connected was to treat with a weak chloride of lime liquor at hand-heat for half an hour and dye with acid colours with addition of formic acid. Colours specially suitable for this purpose were Xylene Light Yellow 2G (S), Orange G, Acid Violet 6BN, Indigo Extract, and Erio Floxine (Gy).

The great majority of articles dyed by the garment dyer were made of wool with a cotton admixture. In his firm's works all items were dry cleaned to ensure freedom from grease. They were then further prepared, as seemed necessary to the dyer, by a boil in water, a mild alkaline treatment with soda or ammonia, or, if necessary, a strip with hydrosulphite. For nearly all goods the wool was dyed acid before the cotton; the cotton was filled in afterwards. With greens, for instance, the wool would be dyed with Algamine Blue and Xylene Light Yellow; or, if very badly faded, with Disulphite Blue and Xylene Light Yellow. Filling-in would be done in some cases with tannin and iron, or with Chlorazol Dark Green B.L. conc. (ICI). Blues would be dyed with Algamine Blue or Alizarine Supra Sky, shaded as necessary with Erio Floxine 2G or Kiton Red; filling in subsequently with Sky Blue FF or Chlorazol Blue 2BS. Red was the one colour which could often be dyed union and using Chloramine Fast Red K (S) and Polar Red 3B (Gy.); but sometimes the fading was so bad that acid dyeing was necessary.

Violets and Blacks

For violets, where fastness to light was the first consideration, a mixture of Erio Floxine 2G (Gy.) and Algamine Blue was used. Where exceptional brilliancy of shade was required, fastness to light must be sacrificed and Acid Violets 4BN and 6BN were used. The cotton was subsequently filled in. Navies were acid dyed first with a mixture of Fast Acid Blue BL (IG), Disulphine Blue, and Acid Violet 6B, the first named colour forming the bulk of the shade; followed by a filling in with a mixture of Direct Black BH and JH and Chloramine Black EX Extra. The dyeing of blacks was always important for the garment dyer and in this case speed was essential. It was quite customary to receive articles up to 12 or even 1 o'clock mid-day, dye them black and have them ready for wear at 5 p.m. The colours used were Acid Milling Black BC (Gy.) for wool and Visco Black (S) and Formic Black MTG in equal quantities for the cotton. The Visco Black was used to give an extra fastness to light. It was rarely necessary to make any departure from this mixture. Occasionally where the article was badly faded it was advisable to dye the wool first with Acid Milling Black in an acid liquor and fill in with a cotton colour afterwards. In nearly all cases, however, the ordinary neutral dyeing mixture would cover fades quite satisfactorily.

Faded Union Material

There were several points in the dyeing of faded union material which were worthy of note. In acid dyeing the wool, some acid colours covered fades better than others, and so a proper choice was essential. In dyeing compound shades with acid colours, fades were covered well if the dye liquor was made up originally to the right tone, so that when the material was dyed deep enough it was on-shade. If this was not done, and considerable shading was necessary with one of the colours, fades then tended to show up.

Cotton colours dyed on wool showed up fades rather badly, and therefore in filling-in the cotton, after acid dyeing the wool, it was important to keep the direct colour off the wool as much as possible. In this connection it was important, first, to choose the direct colour carefully with this property in view; and, secondly, the filling-in liquor must not become acid. When the practice was to use sulphuric acid in dyeing the wool, the filling-in with cotton colours often went wrong; the cotton was thin and too much colour went on the wool, as, in spite of careful rinsing, free sulphuric acid was carried over into the filling-in liquor. The adverse effect was cumulative with further batches of goods. It would be useful to have an easy test for the pH value of the filling-in liquors; the presence of colour making the ordinary tests inapplicable. Since formic acid had been used in the acid dye bath, the filling-in had been more satisfactory, the cotton being fuller, and the wool clearer and brighter. The use of such assistants as Katanol was beneficial also, and allowed the temperature of the filling-in bath to be raised without any fear of the wool being unduly dyed by the cotton colour.

Silk and Cotton Mixtures

Silk and cotton mixtures were quite an important class, as nearly all silk garments had cotton in some part of their construction. With this material the same remarks applied as in the case of wool and cotton, except that fades need not be considered nor was fastness to light so important. Cellulose acetate silk by itself offered no particular problem except that of handling. Some forms of it were exceptionally sensitive to pressure and rubbing, and soon showed glaze marks. Combinations of cellulose acetate with cotton or with wool formed a difficult problem in the early days, but there were now numerous ranges of colours for each fibre. The SRA colours had been found satisfactory.

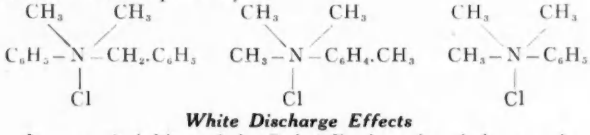
The Use of Leucotropes in Discharge Effects

By F. Farrington

THE origin of leucotropes lies in an attempt to elucidate the constitution of sulphur dyes. The latter were suspected of being complex thiophenols and consequently it should be possible for them to be alkylated or alphylated in neutral or alkaline solution. This was found to be the case and suitable agents were ethyl chloride, benzyl chloride, salts of alkyl sulphuric acid, alkyl sulphates, chlorhydrines, chloracetic acid, mono-chlor-dinitro-benzene and mono-chlor-benzamide.

$$C_6H_5.CH_2\,[Cl + H]\,S.R = HCl + C_6H_5.CH_2.S.R$$

E.P. 21, 898, 1900, describes the application of this reaction to cotton sulphur-dyed fabrics. Thus cotton dyed iwth 10 per cent. Katigen Chrome Brown 5G is treated with a solution in water containing 5 per cent. benzyl chloride and 4 per cent. caustic soda for one hour at 30-40° C., when a pure yellow shade results. Many of the above reagents, however, are inconvenient for commercial usage and attention was turned to quarternary ammonium compounds, many of which can be handled conveniently and will readily decompose, splitting off the required alkyl halide. Suitable compounds are dimethyl-benzyl-phenyl-ammonium chloride, the first of which is known commercially as "leucotrope O." Their formulæ are respectively:—

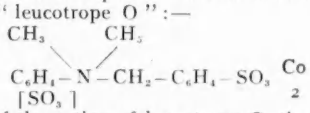


White Discharge Effects

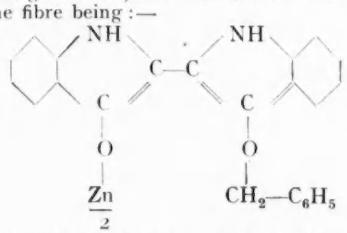
In 1909, Reinking of the B.A.S.F., introduced the reaction for the production of discharge effects on material dyed with sulphur or vat colouring matters. In the latter case, it is the enolic group of the leuco vat dyestuff which is attacked. The compounds formed are sometimes insoluble in acid or alkali and remain fixed on the fibre and coloured discharges may thus be obtained.

When the aralkyl residue which combines with the leuco vat dyestuff contains a salt-forming group, e.g., a sulphonic or carboxylic acid group, the compounds which it forms with the leuco dye are easily soluble in an alkaline bath and can therefore be readily washed away from the fibre, thus giving white discharge effects. Suitable aralkyl compounds are benzyl-chloride-para-sulphonic acid, and the organic ammonium compounds obtained by combining benzyl chloride with dimethyl aniline (yielding leucotrope O) dialkyl-meta-amino-phenol, tetra-methyl-diamino-diphenyl-methane, leuco Malachite Green and leuco Crystal Violet.

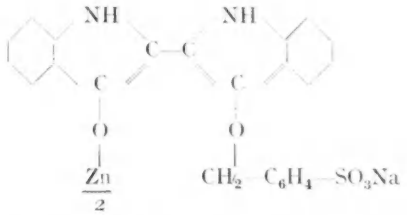
To obtain white discharge effects, the product used in commerce is known as "leucotrope W" and is the calcium salt of disulphonated "leucotrope O":—



As an example of the action of leucotrope O, the following print colour may be applied to Indigo-dyed material: starch-tragacanth thickening (540 grams), Rongalite C (150 grams), water (100 grams), zinc oxide (100 grams), anthraquinone (30 per cent. paste) (40 grams), leucotrope O (70 grams). The fabric is then given a passage in the rapid ager for 3-5 mins. at 216° F., and washed into a hot weakly alkaline bath. An orange discharge results, the formula for the orange compound on the fibre being:—



For a pure white discharge, leucotrope W is employed a suitable recipe being starch thickening (36 grams), Rongalite C (5 grams), leucotrope W (4 grams), zinc oxide (4 grams), anthraquinone (30 per cent. paste) (2 grams), water (5 grams). The indigo is then converted into an orange substance of the formula



which, owing to the sulphonic group, is soluble in alkali and may be washed off the fibre leaving a white effect.

Discharges on Vat-Dyed Grounds

In the case of anthraquinonoid vat dyestuffs, it is necessary to make an addition of strong alkali to the discharge colour. For example, a white discharge on cloth dyed with 5 per cent. Algol Blue K may be obtained by means of British gum thickening (150 grams), water (200 grams), Leucotrope W conc. (100 grams), Rongalite C (150 grams), glycerine (25 grams), caustic soda 35 per cent. (250 grams).

Not all vat dyestuffs can be benzylated readily, and hence it is difficult, in these cases, to obtain satisfactory discharges. This fact may be utilised for the production of coloured discharges on vat dyed grounds. The latter are dyed in easily dischargeable vat colours and printed with a discharge containing a difficultly dischargeable vat colour. Thus a green and orange effect may be obtained by printing on material dyed with Indanthrene Golden Orange G. and adding Caledon Jade Green to the discharge colour. The leucotrope W in the discharge colours benzylates the Indanthrene Golden Orange G very readily, and the product subsequently washes off the fibre, but the Caledon Jade Green is scarcely attacked.

In the preparation of leucotrope O, 34 parts dimethyl-aniline and 17 parts benzyl chloride are heated for 7-8 hours at 60-70° C. The clear liquid solidifies, to a hard crystalline mass, which is then broken up into small pieces and any excess dimethylaniline extracted with ether. The product is soluble in water and alcohol, insoluble in ether and can be recrystallised from a mixture of alcohol and ether. To obtain leucotrope W, 50 parts leucotrope O are stirred into 15 parts 97 per cent. H_2SO_4. The evolved HCl is allowed to escape, dry air passed through the solution to remove any remaining HCl and 85 parts fuming H_2SO_4 (70 per cent SO_3) added gradually, the temperature being maintained at 25-30° C. It is then heated on a water bath for a short time, cooled and poured on to ice; neutralised with lime, evaporated and crystallised. The disulphonic acid derivative crystallised in colourless crystals, insoluble in alcohol. These sulphonic acid derivatives appear to be inner anhydrides between the sulphonic and hydroxyl groups of the ammonium compounds.

Progress of The British Colour Council

350 Enrolments in Ten Months

SPEAKING at a luncheon of the British Colour Council at the Connaught Rooms on January 5, Mr. Malcolm MacDonald, Parliamentary Under-Secretary for the Dominions, and son of the Prime Minister, said he was certain that the future held large increases in Dominion markets for British products. It is hoped, he said, to get something out of the Imperial conferences which would extend imperial trade. The first thing the Dominions would ask, however, was whether we manufactured what they wanted. The British Colour Council was enabling traders everywhere to provide the exact shades required in all materials.

The French and the Americans had provided the colour schemes in former years, but now British industries would know in good time what the season's colours were to be, and that would give them a good start.

Lord Ebury, President of the Council announced that in ten months the Council had enrolled 350 concerns.

Dyes and Dyeing : Technical Progress in 1931

By L. J. Hooley

In common with other industries, dyestuffs manufacture has had to contend with an exceptionally difficult year, but in spite of this it has been successful in maintaining the position built up during the last few years. In striking contrast with the controversy which took place over the renewal of the Dyestuffs (Import) Regulation Act at the end of 1930, has been the agreement as to its renewal for 1932.

THE best indication of the technical progress which the British dye industry is making is to be found in the remarkably long list of new products which have been put on the market, especially by Imperial Chemical Industries, Ltd. Such a list, appearing in such a year, is excellent testimony to the progress which is being made, and is also clear evidence of the determination to supply the user not only with the staple dyes, already established in favour, but also with the continually improving products which can be obtained by systematic and extensive research. As regards the emphasis which has been placed on particular groups of dyes, as shown by research work published during the year, this remains substantially the same as in immediately preceding years, with some slight differences in the amount of attention devoted to particular sub-groups. The main feature of importance in each group will be seen in the reviews of the various sections which follow.

Application

An anonymous paper (*Rev. Gen. Mat. Col.*, 1931, **35**, 201) details the differences in dyeing properties of Bemberg (cuprammonium) silk as compared with cotton and viscose silk, the first named having the greatest affinity for direct dyes. Dyeing at the boil is recommended for level and well penetrated dyeings. H. Mudford (*J. Soc. Dyers and Col.*, 1931, **47**, 222) deals with the dyeing of hosiery composed of mixed fibres and details methods of overcoming the various difficulties which are met in this branch of dyeing. S. R. Trotman and J. E. Bateman describe the production of two-colour effects on mixtures containing viscose silk (*ibid.*, 1931, **47**, 231). R. R. Sleeper (U.S.P. 1,758,877) recommends crude sodium bisulphite (" nitre-cake ") as a cheap substitute for a mixture of Glaubers' salt and sulphuric acid. B.P. 353,512 details methods for dyeing wool with dyes which otherwise level badly. (I.G. Farbenind.) Loaded silk can be dyed in a neutral or fatty soap bath with a suitable metalliferous azo dye. (B.P. 357,400, Soc. Chem. Ind. in Basle). In the ozonisation process for increasing the affinity of animal fibres for dyes, over-ozonisation is prevented by pretreating with formaldehyde (B.P. 345,406, S. W. Wilkinson). Ethanolamines can be added to vat-dye pastes to give improved exhaustion, depth and brightness of shade, and levelling power (U.S.P. 1,782,122, E. I. Dupont de Nemours). For the rapid fixation of chrome colours, the dark green mordant obtained by dissolving five parts of sodium dichromate in eleven parts of hot water, and adding four parts of glycerin, may be used. (V. Borisov and N. Sokolov, *Izvest. Tekstil. Prom.*, 1930, **9**, No. 3, 101). A number of dyes, possibly oxazines, obtained by heating diarylaminobenzoquinones in a high-boiling solvent, can be used for the colouring of paper (B.P. 314,068, I.G. Farbenind.). Fast prints may be obtained on vegetable fibres, by the method used for vat dyes, from certain thiazines. (B.P. 341,119, I.G. Farbenind.). Hydroxy- or amino- anthraquinones or their leuco-compounds are added, with or without hydrotropic agents to vat dye printing pastes (B.P. 349,555 and 350,936, *idem*). Other preparations of vat dye pastes for printing are described by the same applicants in B.P. 318,176, 343,102 and 343,522. Directions are given in B.P. 336,968 for obtaining rapid fast colours upon a vat dye ground (*idem*). Interesting data on Turkey red dyeing are to be found in a paper by L. R. Parks, (*J. Phys. Chem.* 1931, **35**, 488). Several papers and patents have also appeared relative to the production of aniline-black.

Immunisation

A few interesting patents dealing with production of immunised fibres or effect threads are to be recorded. Organic carbimides, especially phenyl carbimides, are now recommended. The fibre is treated dry, in a diluent, and with a catalyst, e.g., zinc chloride, to give products with a nitrogen content of 2.5 per cent. (B.P. 317,019, Soc. Chem. Ind. in Basle). Another treating agent is an alkyl halide, this being used after forming alkali-cellulose; alkyl cellulose is formed, giving resistance to direct dyes, but increased affinity for basic ones. If hydrolysis occurs during storage the products of hydrolysis do not exert any tendering action (B.P. 342,169, *idem*). By using a higher fatty acid chloride in presence of pyridine at 80°, the woven fabric may be immunised (B.P. 343,104, R. Lant and W. Koreska). Effect threads more stable to boiling water than cellulose acetate threads can be made from cellulose nitroacetate. (B.P. 340,501, I.G. Farbenind.) C. E. Mullin reviews the literature upon the production and tinctorial properties of immunised fibres. (*Text. Col.* 1931, **53**, 85 and 5,212).

Acetate Silk : Dyes and Dyeing

In addition to processes dealing with hydrolysis, delustring, weighting and scrooping and the production of matt, cockle and other effects on acetate silk, which space does not permit of reviewing, and which are not all of direct interest in connection with dyeing, there have been quite a number of interesting papers and patents. E. Clayton, from a study of the behaviour of acetate silk with picramic acid, picric acid and 2-amino-anthraquinone, concludes that the primary process is absorption, with perhaps a little adsorption, and that this is followed almost immediately by dissolution. (*J. Soc. Dyers and Col.* 1930, **46**, 367). With basic dyes, K. Kanumaru gives a theoretical explanation of some observed facts, which appear to be consistent with an electrical adsorption theory of dyeing. (*J. Cellulose Inst. Tokyo*, 1031, **7**, Abs. p. 42). Suitable volatile dyes, such as *p*-nitroaniline *p*-xylidine (orange) can be applied in steam (B.P. 349,683, Brit. Celanese). J. M. F. Leaper describes a method of preparing insoluble dyes, by dispersing in a mineral oil compound, which is soluble or dispersible in water (*Text. Col.*, 1931, **53**, 550). L. Clement and C. Riviere (B.P. 350,924) obtain pigment cellulose derivatives by stirring in a liquid medium, which is a solvent for the cellulose derivative. The method is applicable for coloured fibres.

Dyes for acetate silk include (a) *p*-alkoxyaniline → *p*-erisol, which gives greenish-yellow shades, (B.P. 344,884. Soc. Chem. Ind. in Basle), (b) sulphamic acids of 4-amino-1 : 8-naphtholic imide or substituted imide (B.P. 352,099. I.G. Farbenind.) and (c) leuco gallocyanine dyes (B.P. 352,808, Durand and Huguenin). Pyrazolene dyes, which preferably should not contain sulphonic acid groups, are stable to reducing discharges (B.P. 346,751, G. H. Ellis and Brit. Celanese). A number of other patents relating to discharging may be mentioned by number, these being B.P. 315,005, 341,909, 342,333, 348,715, 351,457 and 353,592.

Azo Colours

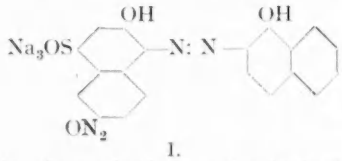
These remain of very great importance, and a large number of patents and some scientific papers have appeared during the year. The main lines of division remain the same as before, with as far as can be seen no specially outstanding developments. Not so much attention has been paid to the technique of diazotisation and the preparation of stable diazo compositions, but the Society of Chemical Industry in Basle, (B.P. 349,339) precipitate diazo compounds as arylsulphonates in presence of a salt of a metal of Group II. Amines containing several acid groups, such as tribromoaniline, can be satisfactorily diazotised by first dissolving in a little pyridine, then adding to a little water containing the nitrate, and then slowly dropping into dilute hydrochloric acid. (S. Krishna and R. L. Bratia, (*Chem. Abs.* 1931, **25**, 298.) A. Hantzsch describes the diazoamino salts in a theoretical paper (*Ber.*, 1930, **63**, 1,786) and J. C. Earl deals with the production of aminoazo compounds from diazoamino ones (*ibid.*, 1930, **63**, 1,666). A. Meyer and R. Vittener (*Compt. rend.* 1931, **193**, 344) have prepared azo dyes from homophthalimide, substantive ones being obtained by coupling tetrazotised benzidine, toluidine or dimethoxybenzidine, on the one hand, with an aminonaphthol or a

naphthol sulphonic acid and the other with homophthalimide. H. T. Bucherer and E. Möhlau have an interesting paper on the *anti*-diazotates and their use in the preparation of azo dyes in substance and on the fibre. Since dyestuff formation takes place in a solution which never becomes acid, it must be due to reaction between the phenol and the *anti*-diazotates, and not the nitrosamine or *syn*-diazotate (*J. pr. Chem.*, 1931, (ii), **131**, 193) orange to brown direct azo dyes are obtained by coupling an arylamine, free from carboxylic and sulphonic acid groups, with an aminobenzoylaminobenzoyl-J-acid as middle component and a sulphoaryl- or carboxyaryl-pyrazolone or indole. (B.P. 336,646, E. I. Dupont de Nemours.) Other direct dyes are those of B.P. 347,742 (Soc. Chem. in Basle), where blues result from coupling a diazo compound having no *o*-or *p*-hydroxyl group with a 1 : 2-aminonaphthol ether or its sulphonic acid and then after again diazotising, coupling with J-acid or an aryl-J-acid.

A further number of patents deal with direct dyes for the level-dyeing or regenerated cellulose silks. These include (a) violet to blue dyes, *p*-aminoazo compound → 1 : 5 : 7-amino naphtholsulphonic acid or N-derivative thereof (B.P. 340,479, R. Brightman and Imperial Chemical Industries), (b) 2-nitro- or 2 : 2¹-dinitro-benzidine, tetrazotised and coupled with the same naphthols as in (a), (B.P. 349,900, *idem*), (c) tetrazotised aminobenzoylphenylenediamine → specified components, (B.P. 338,111, *idem*).

H. Takoaka and H. Yarnauchi (*Proc. World Eng. Congr.*, 1929, **31**, 103) used reduced 6 : 6¹-linitro-di-β-naphthol in azo dyes, and Kalle and Co. (B.P. 341,549) hydroxynaphthylguanidines.

A large number of acid dyes have also been described. A 2 : 5-dialkoxyarylamine diazotised and coupled with a 1-aroylamino-8-naphthol-4 : 6-disulphonic acid gives violets and sulphonated dyes from diazotised negatively substituted *p*-nitroanilines coupled with diphenylamine give browns. (B.P. 334,754 and 339,029, I.G. Farbenind.). A large number of monazo wool dyes contain the sulphonyl group forming part of their structure (B.P. 308,830, 319,585, 337,577, 341,397 and 347,288). P. Ruggli, A. Zimmermann and F. Knapp have assigned the constitution (1) to Erichrome Black, the nitro group occupying position 6 and not as previously believed.



I.

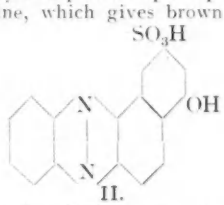
Azo dyes for chrome leather can be obtained by warming an aromatic *N*-nitroso-compound, particularly of an azo dye from a diphenylamine, with an arylamine, phenol or aminophenol (B.P. 338,930, I.G. Farbenind.).

In the chrome and metalliferous dyes the volume of work has not been as large as in some previous years but it is still substantial. A few patents may be mentioned. An aminosulphosalicylic acid may be diazotised and coupled with resorcinal and the product prechromed to give a leather brown (B.P. 352,004, I.G. Farbenind.). Dyes of the type *o*-aminophenol or *o*-aminobenzoic acid *N*-substituted-J-acid are both treated with metallising agents and nitrosated (B.P. 349,304, Soc. Chem. Ind. in Basle). A process of prechroming suitable for certain azo dyes not hitherto applicable, comprises metallisation in an organic base medium (B.P. 335,169).

The ice or development colours, particularly those of naphthol—AS type, have been reviewed so recently (*Dyestuffs Monthly Supplement*, December issue).

Triarylmethane, Basic and Miscellaneous Dyes

Some papers of theoretical interest have appeared on triarylmethane dyes, in connection with the relation between colour and constitution, including one by I. Lifschutz on the constitution of colourless and coloured triphenylmethane derivatives (*Ber.*, 1931, **64**, (B), 161), one by W. Dilthey and R. Dinklage on aniline-blues containing additional phenyl substituents, these being found to exert little influence on the tinctorial properties (*J. pr. Chem.*, 1931, **129**, 24) and one on the constitution of naphthalene-green-V and analogue by F. Frisch (*Helv. Chim. Acta*, 1931, **14**, 669). R.N. Sen and B.N. Sen (*J. Indian Chem. Soc.*, 1930, **7**, 965) prepare triphenylmethane derivatives from quinoline, tretrahydroquinoline, and diphenylamine. Violet triarylmethane dyes are obtained by condensing *m*-2-xylidine with a diaralkyldialkyldiaminobenzhydrol disulphonic acid and oxidising the leuco-compound (B.P. 334,689, E. H. Rodd, F. L. Sharp and Imperial Chem. Industries). Three of four patents on polymethine dyes or intermediates for the same have appeared (B.P. 334,706, 344,409 and 351,555), but otherwise there has been very little published on the other basic colours. In B.P. 335,986 (Soc. Chem. Ind. in Basle) hydroxysulphonaphthaphenazines are coupled with diazo components, examples including 4-hydroxy-2-sulpho-α-naphthaphenazine (II) with diazotised *o*-anisidine, which gives brown shades on wool.

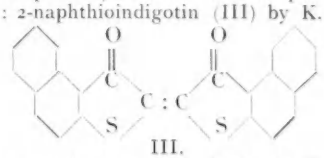


II.

V. G. Gulinov has described a new nitro dye from aminosulpho-salicylic acid (*Ukrain. Chem. J.*, 1930, **5**, (Tech.), 155). Among a number of miscellaneous acid wool dyes, there may be mentioned the yellow-brown to violet-brown dyes made by condensing a 2 :6-diamino-derivative of a sulphonated secondary amine of the benzene series with a 2 : 4 : 6-trinitrobenzene containing a suitable replacable group in position 1. (B.P. 348,902, I. G. Farbenind.); also the yellow and greenish yellow ones obtained by condensing naphthalic or an aminonaphthalic anhydride with a hydroaromatic amine and sulphonating (B.P. 352,139, idem.).

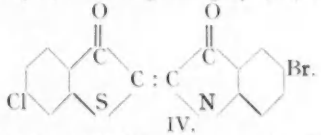
Indigo Dyes and Intermediates

It is rarely that a year's work on indigo fails to contain items of interest and this time there are several. Mention has just been made of the production of sulphur colours by a vapour-phase process, and this can be paralleled by the making of 1 : 2-naphthioindigotin (III) by K. Dziezwonski,



III.

C. Baraniecki, and L. Sternbach (Bull. Acad. Polonaise, *A*, 198), by heating three parts of α-naphthylmethylketone with one part of sulphur at 230°-260°. A convenient method of preparing 5 : 7-dibromoisatin has been described by H. G. Lindwall, J. Bandes, and I. Weinberg (*J. Amer. Chem. Soc.*, 1931, **53**, 317), and J. P. Wibaut and M. C. Georling give a note on the production of isatin from *iso*nitrosoacetanilide 1031, **50**, 41). Naphthalene 1 : 5-dithioglycollic acid gives an cyclisation the violet bis-1 : 2-naphthothioindigotin-5 : 5¹-dithioglycollic acid (A. Corbellini and L. Albenga, *Gazzetta*, 1931, **61**, 111). Of interest are the anomalous stable reduction products, differing from the leuco vat dyes, which are obtained from certain substituted thioindigos, carrying halogen and methyl substituents, e.g., the product, insoluble in cold caustic alkali, and useful for printing, obtained by hydrogenating 6 : 6¹-dihalogeno-4 : 4¹-dimethylthioindigos in aqueous alkaline suspension in the presence of a nickel catalyst. (B.P. 334,878, 234,919-20, 338,104, 350,862, 352,283, I. G. Farbenind.) High claims have been made for a number of new indigo dyes, including the grey (IV) obtained in B.P.



IV.

341,224 (I. G. Farbenind.). The two dyes having a methyl group in place of the chlorine and bromine or chlorine in the same position as the bromine in (IV) are also greys (B.P. 345,130, *idem.*) the pyrazolethioindigos of B.P. 345,349 (*idem.*), are also greys. New greens and greenish-blues have also been described (B.P. 313,493, 335,043, *idem.*).

(*To be continued.*)

Cutting Costs in the Dyehouse

Some Points from American Practice

THE reduction of costs in the dyehouse was discussed from many angles in a recent issue of our contemporary, the *American Dyestuffs Reporter*. Under the heading of dyestuffs and chemicals, the author of the article, Mr. H. H. Field, said one of the most important things to consider is the proper selection of these materials for your own particular use, and your various methods of application. For example, using sulphur colours as illustration, on piece dyeing it is of utmost importance that colours which will exhaust uniformly and have a strong exhaust be used. If dyestuffs and chemicals are selected with an eye to such requirements as just referred to, and to the many other requirements these materials should meet, much money can be saved for the mill. However, if they are selected without thorough tests being run, much money can be needlessly lost.

The Function of the Laboratory

A well equipped laboratory with a competent man in charge will pay for itself in a short while, and will mean a large saving to any mill regardless of size, if run on a scientific scale. In this laboratory, product samples of the different dyestuff and chemicals concerns can be kept, and when desired, tests may be run to determine money value and quality of dyes, fastness, exhaust, effect of after-treatment, change of ageing, etc. This abolishes to a great extent the very costly method of running tests on a large scale in the mill on products of which the quality is unknown. The varying conditions from laboratory to mill make it necessary that these large scale tests be run at times to make sure that the product will meet your local conditions. However, the laboratory can weed out the products which will not be satisfactory at all, and these mill-tests need only be run on the products that from the laboratory test show promise of value to the mill. The mill laboratory will do much to reduce the cost on dyestuffs and chemicals.

The equipment of the dyehouse is naturally a large item in the reduction of cost. There is much food for thought in the upkeep of the equipment, and the adding to the machines of any devices which will be labour savers and will improve quality, such as air and electric hoists, etc. There are numerous devices of this kind which, if adapted to the mills needs will be a big item in cost reduction. The actual upkeep of equipment is an item which will determine to a huge extent the amount of saving in your dyehouse. If equipment is kept in good running condition at all times the chances of breakdowns and poor work are decreased greatly. If upkeep is neglected only poor work and costly losses of time due to breakdowns can be expected. This applies especially to operations such as piece dyeing, resizing, etc. Unless the rolls on the machines are kept in perfect alignment, and the surface of the rolls smooth, the result will be torn and wrinkled cloth, and uneven dyeing.

Importance of Cleanliness

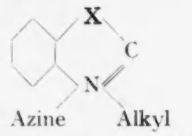
Cleanliness should also be a big item in every dyehouse, for it goes hand in hand with cost reduction. Even when running the operators should be on the alert and prepared to clean up in any spare moments they may have. A good policy is to cease production at the end of the week early enough to get a good clean-up from top to bottom. Much material can be ruined through failure to keep the equipment and plant, as a whole, clean.

The reduction and control of labour cost is a much more difficult problem than it seems to be without examination, especially so in dyeing. In the dyehouse the operator must be a man of good intelligence, and must be well acquainted with his job. Therefore, the idea of replacing trained men by cheaper and untrained men is not logical in the dyehouse. Of course, these untrained men have as much intelligence as the trained worker, but the cost of training him and the cost of his mistakes will overshadow the saving in the employment of the untrained man. Reduction of labour cost requires systematic and well worked out plans. Nothing can be more detrimental to quality and production than a haphazard attempt at labour cost reductions, and with a sacrifice of production, cost cannot go down, and with quality impaired, the cost is greater than the gain.

Dyes of the Polymethine Series

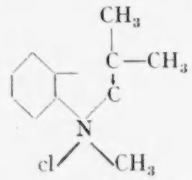
Recent Developments

THE dyestuffs from cyclic ammonium bases such as those designated by the formula :—



where X is a divalent residue, such as CH_2, S, CH=CH, and z a substituent such as halogen or alkoxy which can be prepared by the otherwise general method applicable to a cyclic ammonium salt of an inorganic acid containing in α or γ position a reactive methyl group, namely by treating with an orthoformic acid ester or a salt of formic acid, are now obtained along with others by condensing with formic acid or a salt thereof instead of a cyclic salt containing a reactive group in the α or γ position, the corresponding methylene base or cyclic salt of an organic acid (B.P. 328,357, R. Kuhn and A. Winterstein).

Another process for making dyes of the polymethine series consists in taking an ammonium salt having a reactive α-methyl group, or its corresponding pseudo base, first condensing with a compound X. CH : NH (e.g., formiminochloride) to give an intediate as shown under :—



and then condensing with a second molecule of the same or a different cyclic ammonium salt (B.P. 334,206, S.C.I.B.)

Intermediate products for this series are obtained in B.P. 362,174 by causing two molecular proportions of a 1 : 3 : 3-trimethylindolenium salt having a reactive group in α position with one molecule of formaldehyde.

United States Chemical Exports

Notable Gains in Newer Products

EXPORT gains were made for certain newer American chemicals during the first nine months of 1931 according to figures prepared by the Chemical Division of the United States Department of Commerce. Nearly 50 commodities recorded increases compared with corresponding nine months of 1930, although total exports fell 24 per cent. in value, from $134,219,000 to $101,883,000.

With the discovery of the suitability of calcium chloride in road work, and also for minor specific purposes, exports of this product have been gaining steadily and during the current three-quarter period exceeded $534,000 (46,000,000 lb). Exports of miscellaneous agricultural insecticides, household disinfectants, deodorants, and similar compounds; acetone; miscellaneous synthetic organic chemicals; nitro-cellulose and acetocellulose solutions; and lacquers—all in the category of newer American commodities—expanded during the period under discussion. Other industrial chemicals in which undoubtedly the marked fall in price was a contributing cause to greater business were copper sulphate, which doubled in quantities shipped, and sodium cyanide, sodium sulphate, caustic, tin compounds, chlorine, and gases other than ammonia. Sodium borate, however, recorded an increase in value to $2,497,000, but a small loss in quantity to 126,000,000 lb.

Five per cent. more carbon black, or a total of 68,900,000 lb., were exported during the nine-month period of the current year, although values declined 20 per cent. to $3,700,000.

Butanol Manufacture Contemplated in Japan.

JAPANESE engineers are reported to be engaged in research work on butanol with a view to manufacturing the commodity on a commercial scale in the near future. A small quantity of butanol is now imported into Japan from the United States and Germany.

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.*

Dearer Dyestuffs

A number of advances in the prices of dyestuffs have been announced during the past month. In some cases the increase has been as much as 40 per cent., but, taking a general survey, the average rise has been something of the order of less than 20 per cent. On the manufacturers' side, the revision of prices has been defended on the grounds that the depreciation of the value of sterling has increased the cost of raw materials, that the price of benzol in this country is much higher than it is abroad, and that the manufacture of some dyes involves extremely long, complicated and costly processes. Moreover, they state that low dye prices are disadvantageous to the consumer, for the reason that unless there is a reasonable margin of profit on his products the dye manufacturer cannot put aside money for research, neither can he publish a continuous stream of useful pattern cards and provide technical information and assistance. While a number of dyers have accepted the increase as inevitable, some have entered vigorous protests, and have suggested that the makers are running near the profiteering line by increasing the prices to the extent they have done. The cost of production, they point out, has not materially increased, and the makers are certainly not helping the country to increase the export trade in textiles. In some quarters it is urged that dyestuffs manufacturers have for some time been operating on an uneconomic basis, and that the price revision is nothing more than evidence that they feel the time has arrived to make a reasonable living.

British Colours for Spring

The British Colour Council has issued four cards, for hosiery, silk, wool, and leather, showing colours for the spring season of this year. These attractive productions give a full range of the fashionable shades which will be offered at the opening of the season, and should be a reliable guide to distributors and producers. Blue, beige, and orange reds are the leading colours, and provision is also made for the demand for grey which is expected. Two features which are forecast are a general lightening in the tone of materials used for women's wear and the substitution of the vogue for contrasting colours by one for matching or harmonious toning. Due consideration of this fact was made when the various colours were created and issued to members of the Council last August, at which time it was stated that "for the spring, bright colours, lightening in tone, would be greatly used, while there would be a come-back of beige and grey." Reports from Paris now indicate that fashions there are in conformity with the British Colour Council's earlier intimation, showing that Paris is actually following Britain's lead. These colours include sapphire, lapis lazuli, moonstone, and coral reef, while snow shadow, valley mist, and golden glow are among the fanciful names given to shades of the predominant colours. These colours, it may be added, should continue in favour until late autumn.

Colour Standards in the United States

A clearing house of reliable information on colour for artists and industrialists is being organized by thirteen co-operating scientific and technical societies in the United States. This new Inter-Society Colour Council, whose chairman is Professor E. M. Gathercoal of the University of Illinois, will standardize the names, measurements, and specifications of colours. Practical men confronted with colour problems will, according to the plans of the council, be put immediately in touch with those who have appropriate technical knowledge. Duplication of effort and loss of time will thus, it is hoped, be avoided. At present there is a gap between those who have the scientific knowledge of colour, such as physicists of the Optical Society of America or textile chemists, and those faced with practical problems in the use of colours. The Colour Council, made up of delegates from the constituent societies, and of private persons interested in the same programme, will set about filling this gap. At the same time the Council will, by the spread of popularized information, promote a wider understanding of colour problems. The co-operating bodies are the American Association of Textile Chemists and Colorists, the American Chemical Society, the American Oil Chemists' Society, the American Pharmaceutical Association, the American Society for Testing Materials, the Illuminating Engineering Society, the International Society of Master Painters and Decorators, the National Academy of Design, New York Museum of Science and Industry, the Optical Society of America, the Technical Association of the Pulp and Paper Industries, and the Textile Colour Card Association.

American Colour Chart

The colour correlation chart for spring and summer, 1932, has just been issued to members of the Textile Colour Card Association of the United States. In this chart, which will serve as a colour guide in co-ordinating apparel and accessories, the fashionable shades for town, sports and evening costumes are listed according to their cast, and for each group are given the correct colours for shoes, bags, hosiery and gloves. The fashion highlights in colour harmonies and contrasts, including bi-colour and tri-colour effects, are indicated in another division of the chart, which is to be used in conjunction with the 1932 spring season cards for silks, woollens, shoe and leather and hosiery issued by the Textile Colour Card Association. The chart will not only serve as a merchandising reference for retailers to indicate the outstanding shades for accessories, but it will also provide a guide for manufacturers in keying their production to the basic costume colours for the coming spring and summer.

The British Industries Fair

The British Industries Fair, which will open at London and Birmingham on February 22, is expected to be the greatest industrial exhibition ever held in any country. About 120,000 invitations to buyers all over the world have been sent out, and the response has been very encouraging. Home buyers will undoubtedly come to the Fair in large numbers, and the textile industry will be one of the chief trades to benefit from their visits. All the textile exhibits will be concentrated in one building, instead of being distributed between Olympia, the White City and the Albert Hall, as last year. Every description of textile material will be displayed—cotton, rayon, silk, wool and linen. Dyers will be well represented, the leading firms having taken a considerable amount of space, where the skill and inventiveness of British dyers and finishers will be in evidence. The advance that Great Britain has

made as a manufacturer of dyestuffs during recent years, especially during 1931, will be shown on Stand No. B71/74 at Olympia. This exhibit will show that during 1931 no less than 200 new products have been produced by the dyestuffs group of Imperial Chemical Industries, Ltd. All types of dyestuffs will be represented among the new products, and, in view of the present tendency for faster shades, not only on high-class articles but also on materials of moderate quality, it is encouraging to note that manufacturers are fully alive to the requirements of the moment, and 30 of the 200 new dyestuffs are claimed to produce shades fully suitable for use in the manufacture of "guaranteed fabrics."

Two New I.C.I. Colours

Monolite Red TS is a new addition to the range of Monolite colours made by Imperial Chemical Industries, Ltd. It is distinguishable from the other colours in the range in that it possesses a very high degree of transparency. It will, therefore, be found extremely useful where the employment of the usual Monolite Red type of pigment colour is prohibited on account of opacity or covering power. In shade and general fastness properties this colour closely resembles a toner lake of Monolite Red CS, but unlike the latter product possesses very good transparency. Its chief outlet will be for the manufacture of certain classes of printing inks of good fastness to light, for use in three-colour printing, tinplate printing, and wherever a high degree of transparency is an essential property. In addition, it should find use in the manufacture of varnishes and certain types of cellulose lacquers. It is insoluble in water and requires no precipitating agent. The colour is simply ground with or without a substratum into the required medium, and is ready for use. Monolite Red TS is unaffected by acids; in contact with alkalies or lime, the shade turns a little yellower.

CALEDON DARK BLUE 2RDS is an addition to the range of Caledon dyestuffs. It is a red shade navy blue suitable for application on all types of cotton goods, either in the open beck or in machines, and is particularly recommended for the dyeing of piece goods on the jigger. It is also suitable for the dyeing of viscose, linen and silk. In combination with Caledon Dark Blue GS, the greenest navy blue in this I.C.I. range, all tones of navy blue may be produced. The new colour is dyed according to the process given as Method I in the I.C.I. pattern card on Caledon and Durindone colours. In printing, it is suitable for application by the potash-formosul method.

Dyestuffs Licences for January

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during January has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee. The total number of applications received during the month was 382, of which 290 were from merchants or importers. To these should be added two cases outstanding on December 31, making a total for the month of 384. These were dealt with as follows:—Granted, 371 (of which 362 were dealt with within seven days of receipt); referred to British makers of similar products, seven (all dealt with within seven days of receipt); outstanding on January 30, six. Of the total of 384 applications received, 369, or 96 per cent., were dealt with within seven days of receipt.

Beam Dyeing with Vat Colours

Avoiding Damage to the Dyed Yarn

THE attractive proposition offered by the dyeing of cotton yarns in package form were discussed by Mr. J. R. S. Goodall in the course of a paper read before the Manchester Section of the Society of Dyers and Colourists, on January 15, Mr. F. Scholefield presiding.

Mr. Goodall said the chief advantages were small labour charges and minimum space requirements, economical consumption of dyestuffs, and a resulting dyed yarn which had not been handled in process, and consequently was free from roughness or breakages. The latter point resulted in a great saving of time in preparation, and considerable assistance to loom production as against other methods of dyeing warp yarns.

A 4-beam dyeing plant capable of holding 880 lbs. of yarn, had turned out 8,800 lbs. of vat-dyed yarns per day of 10 hours, using the labour of two men. Such results were naturally desirable, but unfortunately there were many difficulties in the way of their being generally obtained. The chief of these were those of obtaining level results, and clean fast colours without a tendency to rub, and were due to a number of factors, commencing firstly with imperfectly constructed beams with a liability to channelling. This admitted free passage of dye liquor through the opened parts of the beam, and consequently there were darker shades on contact threads than on the bulk of the unopened yarn. This was particularly difficult to prevent at the end of the beams, as the slightest deviation from the perpendicular of the beam flanges also invariably gave varying densities of yarn in this part. A method used for the prevention of this trouble in which the yarn was cross wound on beams with sliding and locking flanges, the flange being pressed up to the yarn after beaming and then locked in position, had been used with some success, more particularly with soft fairly wound beams.

Simplifying the Dyeing Process

This fundamentally important condition of beam construction had long occupied the attention of many textile engineers, and patents for achieving satisfactory results had been taken out, notably by Brandwood with his patented density controlling apparatus. The principle of comparatively soft winding and then compressing to an even density throughout the whole mass of the yarn, was being most successfully used in package dyeing of various kinds, but so far owing to subsequent winding difficulties had not been used to any extent in beam dyeing. Once there was a well-constructed beam the first difficulties were solved. It would then be possible to deal with the other troubles such as unsuitability of dyestuffs for the purpose, for anthraquinone vat dyestuffs as a class were by no means perfect for the process under consideration. Groups of indigoid and carbazol vat dyestuffs, such as the durindones, ciba, algol, thio-indigo, helindones, etc., could easily be dyed with far less difficulty than the faster anthraquinone derivatives—the caledon, indanthrene, cibanone, alizanthrene, anthrene, ponceau, and solanthrene types, though there were exceptions to both general rules. Individual vat dyers on beams varied considerably in the results they obtained for the same type of colour, but not even the best of them would be prepared to say that he would undertake the dyeing of complete ranges of anthraquinone vat shades.

The process of beam dyeing is in fairly general use in certain countries, notably Holland and Germany, but a true story of the experiences of most of the users would be a deterrent to many people who were considering undertaking this, which is probably the most difficult job in fast colour dyeing. Recently, however, dye manufacturers had recognised certain essential properties of anthraquinone vat dyestuffs for use in packing dyeing, such as (1) complete solubility, even in highly concentrated solutions; (2) that such solutions must be free flowing, and not viscose; (3) that they must be completely free from insoluble foreign matter; (4) that they must not form complex colloidal precipitate when fatty bodies were scoured out from cotton during the alkaline scouring which occurred during the circulation of the vat dyebath; (5) solutions must remain unaffected by the application of pressure; and (6) the rate of dyeing must be sufficiently slow to allow the dye solution to be in contact with the whole of the material to be dyed before any substantial amount of colour was taken up by the fibre.

Dyes and Dyeing : Technical Progress in 1931

By L. J. Hooley

The following review of technical progress in dyes and dyeing in 1931 is continued from "The Chemical Age" Dyestuffs Monthly Supplement of January 9 (pages 4 and 5).

THERE is little to report regarding sulphur dyes. They are notably obtainable from a very wide choice of starting materials and now G. D. Palmer, jun., and S. J. Lloyd (*J. Amer. Chem. Soc.*, 1930, **52**, 3388), have shown that practically all hydrogen-containing organic compounds, will give sulphur colours when vapourised and passed over or through molten sulphur above 300°. Alcohol yields a grey-brown and aniline a green.

Hydroxylated phenazines may be thionated in presence of molybdenum compounds, thus from 3 : 7-aminohydroxyphenazine, dissolved in caustic soda, and heated with sodium polysulphide and molybdic acid, a clear bluish-bordeaux-red is obtained. (B.P. 335,297, Chem. Works from Sandoz.) Bluish-green sulphide dyes are obtained by sulphurising a 6-arylamino-2-*p*-hydroxyarylaminonaphthalene; the first named may be prepared by condensing a 6-arylamino-β-naphthol with a *p*-aminophenol (B.P. 336,061, *idem.*). Alkylation and aralkylation of sulphur colours on the fibre is applicable to sulphurised tetra- and penta-chlorinated dinaphthylene dioxide (B.P. 337,061, I. G. Farbenind.).

Spent liquors from sulphur black dyebaths can be clarified before discharge to the drain, by treatment with ferric chloride, preferably with addition of sufficient hydrochloric acid to give a final pH value of 6.6-7.7 (W. Rudolfs and W. H. Baumgartner, *Ind. Eng. Chem.*, 1931, **23**, 906).

Anthraquinone Intermediates and Dyes

Excluding the anthraquinone vat dyes, which will be dealt with separately in the following, a large number of patents and scientific papers have appeared during the year. Many of them are unsuitable for brief individual description, but several groups may be mentioned; these include (a) quite a variety of syntheses by the *o*-benzoylbenzoic acid and similar routes, (b) a number of anthrone derivatives, (c) reduction products of anthraquinone derivatives carrying carboxylic groups or other groups attached through a carbonyl, and (d) several derivatives of hydrocarbons analogous to anthracene, but containing additional fused rings as in benzanthracene. One or two matters of more immediate technical interest may be reviewed shortly. C. J. Thatcher (U.S. Patent 1,782,488) purifies 30 per cent anthracene by grinding in a ball mill with a solvent such as benzene or carbon tetrachloride, which will extract phenanthrene, oils, etc. V. I. Minaev and B. P. Federov have investigated the yields in the various stages in the production of alizarine from anthracene by Perkin's method; these being 70 per cent. of *ms*-dichloroanthracene, 81-5 per cent. of sodium *ms*-dichloroanthracene-2-sulphonate, 82 per cent. of anthraquinone-2-sulphonic acid, and 90 per cent. of alizarine. (*Rco. gen. mat. Col.*, 1930, **34**, 330 and 376.) K. Lauer has made extensive investigations into the anthraquinone sulphonic acids (*J. pr. Chem.*, 1931, (ii), **130**, 185). Halogenated phthalic anhydrides are condensed with halogenated phenols in sulphuric acid with boric acid to give 6-chloro-, 5 : 6- and 6 : 7-dichloro- and 2 : 6 : 7-trichloro-quinizarine (B.P. 339,589, P. F. Bangham, L. J. Hooley, J. Thomas, and Scottish Dyes). A number of new derivatives for dyeing acetate silk have been described including anthraquinone carboxylic amides (B.P. 350,928, I. G. Farbenind.). Amino or alkylamino-dianthraquinonylamines are sulphonated with oleum in presence of boric acid and a mercury catalyst to give wool dyes (B.P. 349,365, *idem.*). Other blue and blue-green wool dyes, these being principally 1 : 2 : 4-derivatives have also been described, including these obtained by condensing 4-bromo-1-aminoanthraquinone-2-sulphonic acid with an amino-aryl ether of a polyhydric alcohol (B.P. 339,266, *idem.*).

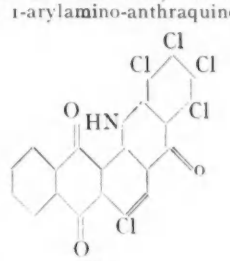
Indanthrone, Flavanthrone, and Pyranthrone

Indanthrone derivatives can be obtained by condensing aminoanthraquinone with *o*-halogenonitro-*o*-benzoylbenzoic acid, reducing with formation of tetrahydrodiphenazine and then cyclising through the carboxylic group (B.P. 334,872, W. Smith, S. G. Willimott, J. Thomas, and Scottish Dyes).

In connection with the production of indanthrone by alkali-fusion of 2-aminoanthraquinone, I. Maki has examined the effect of the addition of phenol, as well as that of other factors such time and temperature (*J. Soc. Chem. Ind., Japan.*, 1930, **33**, 456B). As is known, aldehydes will condense with the imino-groups of indanthrone to give products greener than the parent substance. These condensation products have now been submitted to further treatments, including purification from sulphuric acid, halogenation, and heating in inert media for the production of further indanthrone dyes (B.P. 343,994, 346,328, 344,777, 348,170, and 351,032, I. G. Farbenind.). It is interesting to note that the green-yellow body obtained by treating indanthrone with sulphuric acid and chromic acid and thought to be the azhydrine, obtained by joining two indanthrones through a single nitrogen atom in each, has been separated by R. Scholl and H. Lamprecht into the azine and dihydroazine forms of indanthrone (*Ber.*, 1930, **63**, 2126). A new sulphuric ester of indanthrone, tetrahydrodianthraquinoneazine tetrasulphuric ester has been described (B.P. 334,902 and 334,921, D. A. W. Fairweather, J. Thomas and Scottish Dyes).

Flavanthrone can be obtained directly from 2 : 2¹-dichloro-1 : 1¹-dianthraquinonyl by heating with ammonia; other products can be got from amines, phenols or mercaptans and the same or similar 2 : 2¹ disubstituted 1 : 1-dianthraquinonyls (B. P. 336,983, W. Smith, L. J. Hooley and Scottish Dyes).

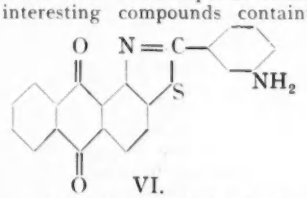
A variety of new halogenated anthraquinoneacridones have been prepared of which (V) may be given as an example (B.P. 338,182 and 338,216, I. G. Farbenind.). As intermediates for similar acridones, a 1-arylamino-anthraquinone carrying an



V.

aldehyde-group. *ortho* to the imino group, may be used, being cyclised with halogenating agents (B.P. 350,501, *idem.*). Acridones, more complex than the ones already named, can be synthesised by condensing an *o*-amino-anthraquinonecarboxylic ester or amide with a halogenated polynuclear compound having at least four rings, such as dibenzpyrenequinone (B.P. 346,677, *idem.*).

Vat dyes with structures resembling doubled benzoylaminoanthraquinones can be obtained by condensing two molecules of an α-amino-anthraquinone with one molecule of a symmetrical dicarboxylic chloride, such as diphenylmethane dicarboxylic chloride (B.P. 339,267, *idem.*). Other acylamino-derivatives are obtained by condensing aminoanthraquinones with halogenated pyrimidines and aroylating (B.P. 345,982, H. A. Piggot, W. W. Tatum and Imperial Chem. Industries). A number of interesting compounds containing sulphur,



VI.

chiefly thiazoles have been described; P. Ruggli and W. Heitz (*Helv. Chim. Acta*, 1931, **14**, 275) have cyclised the products from *m*-nitrobenzoylchloride and 1-amino-2-mercapto-anthraquinone to obtain *m*-aminophenylanthraquinone-2 : 1-thiozole (VI).

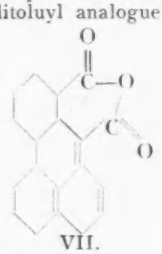
Benzanthrone Dyes

The benzanthrone series is of most interest as a source of violet, blue, green, and black vat dyes. These dyes usually contain either the dibenzanthrone or *iso*-dibenzanthrone ring structure, and this year interest centres round the production of halogen derivatives, which are generally blue, and other derivatives, often containing nitrogen, which are blacks, and these are obtained by treatment of other dyestuffs of the series. There has not been the same attention paid to the synthesis of dibenzanthronyls, dibenzanthrone and substituted dibenzanthrones from intermediates containing fewer rings. Like the indigos and indanthrones, the dibenzanthrones can be improved for many purposes by halogenation, and the various halogenated derivatives differ considerably amongst themselves in tinctorial properties, so that the method chosen for their preparation often exercised a considerable influence on the final properties. The newer products include dyes which contain both chlorine and bromine (B.P. 340,262, 347,234, and 351,948, I. G. Farbenindustrie). Metallic chlorides such as those of iron, chromium and copper have been used, in the presence of aluminium chloride (B.P. 350,457, Aziendi Chimica Nazionali Associate).

Another vat black obtained by using hydroxylamine is described in B.P. 348,159 (S. Thornley, J. Thomas and Scottish Dyes) a sulphonated dibenzanthrone being treated with hydroxylamine and ferrous sulphate and then alkylated, if desired. Soluble sulphuric esters of dibenzanthrone give blacks on sufficiently strong oxidation (B.P. 339,324, R. S. Barnes, R. Fraser Thomson, J. Thomas and Scottish Dyes). It will be remembered that the first benzanthrone derivatives were prepared by the Skraup synthesis, from aminoanthraquinones. Some new pyridinobenzanthrones have now been prepared from 9- and 11-aminobenzanthrones, and these also give blue vat dyes on alkali fusion. Quinolines from other polycamines, such as amino-*allo*-*ms*-naphthadianthrones, have also been prepared (B.P. 349,059, I. G. Farbenind.). Heating dibenzanthrone with phosphorus sulphide at 250°-300° gives a red dye (B.P. 344,336, C. Shaw, J. Thomas and Scottish Dyes).

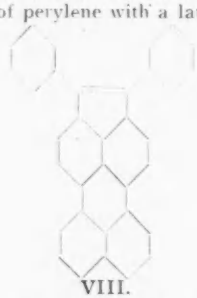
Perylene Dyes and Derivatives

Some further derivatives such as 3 : 9- and 3 : 10-dibenzoylperylenes, bodies which give dibenzanthrones on further condensation have been prepared. Neither the di-*m*-and di-*p*-toluylperylenes, obtained by condensing perylene with *m*- and *p*-toluyl chloride in presence of aluminium chloride, nor the corresponding *o*-ditoluyl analogue is identical with the



VII.

compound obtained from toluene and perylene-3 : 9-dicarboxylic chloride (A. Pongratz, and A. Halabarda, *Monatsh.*, 1930, **56**, 163). When oxides with dilute chromic acid, rupture takes place with formation of a dicarboxylic anhydride (VII). Treatment of perylene with a large excess of benzoyl

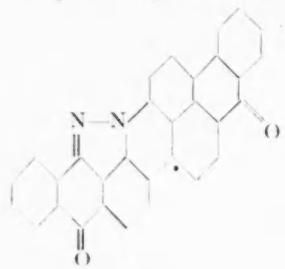


VIII.

chloride in the presence of aluminium chloride gives some 3 : 4-dibenzoylperylene in addition to the previously recognised 3 : 9-isomeride, and this reduces to the new hydrocarbon diphenylaceperylene (VIII) (A. Ziuke, and O. Berndorf, *ibid.*, 1930, **56**, 152). Aminoperylenes condense with perylene dicarboxylic chlorides giving dyes analogous to the benzoylaminoanthraquinones a deep brown being obtained from diaminoperylene-3 : 10-quinone and perylene-3 : 9-dicarboxylic chloride (B.P. 347,099, F. Bensa). Fifteen perylene vat colours have been described in a paper by E. Vecchiono, and G. Matscher (*Chem. abs.*, 1930, **24**, 5161 .

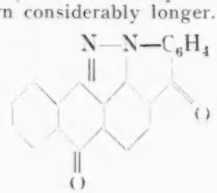
Miscellaneous Complex Vat Dyes

While the majority of the anthraquinone vat dyes are symmetrical in structure, in the sense of containing two equal halves, during the last two or three years there have been quite a number of new bodies synthesised, which contain a skeletal structure, as if a half of one dye had been joined to a half of another one; as in the benzanthronylpyrazolanthrones, of which the parent body is IX. The resemblance to



IX.

dibenzanthrone will be evident. In B.P. 344,057, and 345,651, a variety of derivatives of this parent body are prepared which contain substituted amino-groups, hydroxy, or thiol groups. A new product of even more complicated structure and containing probably three different parts corresponding to the two different ones in the benzanthrone pyrazolanthrone is obtained by substituting the last mentioned with an anthraquinonylamine acid then fusing with alkali (B.P. 345,728, I. G. Farbenind.). Somewhat similar are the pyrazolanthrone acridones (X). One way of preparing these is from a pyrazolanthrone and an *o*-halogenoaldehyde, with alkali-fusion to give the acridonic (B.P. 344,558, I. G. Farbenind.). The anthraquinonylaminobenzanthrones have been known considerably longer. Several new deriva-



X.

tives have been described (B.P. 337,741, 341,804, 348,706, 349,115, *idem*.). For typical examples of other highly complex vat dyes, which cannot be referred to shortly, the following patents may be referred to, B.P. 340,334, 340,524, 347,100, 347,236, and 348,849, 350,575, I. G. Farbenind.).

Scottish Dyestuffs Market

THERE has not been much to note during the past month. The new tariffs are expected to improve the general position, but there has not been much change so far. The outlook, however, is considered to be better. In Dunfermline, the linen industry has been showing signs of brightening and the depreciation of sterling has itself acted as a safeguard on various lines not yet covered by the anti-dumping duties. The outlook for jute is also considered rather better on the whole. At a meeting at Paton and Baldwin's on Friday it was resolved that the issued preference and ordinary shares should be converted to stock, this being transferable in multiples of £1. It has been decided to re-open the British Silk Dyeing Co.'s factory at Balloch, Loch Lomond. Last December, a Swiss group acquired an interest in the company and new and additional machinery is to be installed. It is thought that the situation has been brought about primarily as a result of the silk duties and the tariff prospects. Several hundreds of men and women in the Vale of Leven should obtain employment in consequence.

Dyes and Textiles

Notes on Current Progress and Research

IN many coloured materials, both knitted and woven, consisting of cotton and Bemberg silk the appearance is marred by a difference in shade between the cotton and the silk. The dyeing affinities of these two fibres is so different that dyers find it exceedingly difficult to secure equality of shade. Generally, dyers find it advisable to dye at the boil in their endeavour to produce equal depths of shade on both fibres with direct dyes, but complete success is not often attained. Recently the American Bemberg Corporation, Ltd. (U.S. Pat. 1,797,247) have described a method whereby these difficulties can be largely avoided.

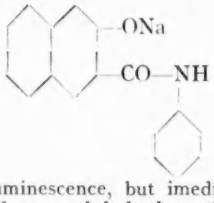
It is suggested that before weaving or knitting, the cotton yarn be subjected to a mild treatment with a cuprammonium solution. This has the effect of swelling the cotton and raising its affinity for direct dyes to equal that of Bemberg silk. It will be remembered that this type of silk is manufactured from a cuprammonium solution of cotton, and hence the suitability of the cuprammonium treatment for the cotton yarn. The pre-treatment is effected in an ordinary hank mercerising machine and is actually carried out in two stages. In the first, the cotton yarn is exposed to the action of a somewhat dilute solution containing 0.39 to 0.40 per cent. of copper (together with ammonia as solvent); in the second stage a stronger solution containing 0.70 to 0.75 per cent. of copper is employed. In these treatments the yarn tends to shrink in length and this may be counteracted by suitable stretching. Residual copper is removed from the cotton yarn by washing with an ammoniacal solution of ammonium sulphate (2.5 litres of ammonia sp. gr. 0.901, and 0.5 litres of sulphuric acid sp. gr. 1.701, per 100 litres of water) and then with dilute sulphuric acid. It is obvious that the washings from the second stage may be used again for the first stage.

Effect of pH on Detergent Power

The detergent power of a soap solution is not easy to measure. Many workers have assumed that this detergent power rises parallel with the decrease in surface tension of the soap solution as produced by additions of alkalis or other substances, but this view is not necessarily correct. In their investigations, Rhodes and Bascom have therefore approached the subject more directly and compared the detergent properties of the various soap liquors used by actual washing (under standard conditions) of artificially soiled cotton fabric and determining the resulting whiteness. In these tests a 0.25 per cent. solution of soap flakes was employed throughout. From the results it is evident that the addition of an alkali may not always increase the detergent power of a soap solution. In fact the addition may function in the opposite manner. For example, all additions of borax decreased the detergent power. But with the addition of other alkalis it became apparent that an advantage was obtained up to a certain optimum maximum pH and then with further additions the detergent properties were adversely affected. The optimum pH is 10.7, and to secure this it is necessary to add 0.6 per cent. of caustic soda, or 6.4 per cent. of sodium phosphate, or 16.2 per cent of soda ash.

Naphthol Dyes in Ultra-Violet Light

According to O. Mecheels (*Textilber.*, 1931, *12*, 581) many useful facts may be discovered by examining Naphthol AS solutions, and cotton fabrics impregnated with this substance and dyes derived from it, by the aid of ultra-violet light. It appears that Naphthol AS when in the form of its sodium or potassium salt having the chemical structure,



shows a yellowish luminescence, but imediately the salt is converted into the free naphthol then this luminescence disappears. All aqueous solutions of Naphthol AS prepared with caustic soda or potash show a yellow luminescence when viewed by reflected or transmitted ultra-violet light, and when these solutions are dried into cotton fabric this also shows a similar luminescence. Ammonia apears to have scarcely sufficient alkaline power to convert the naphthol into a salt form showing this characteristic luminescence, and if an ammoniacal solution of Naphthol AS is spotted on fabric this has but a bluish white luminescence.

In large scale dyeing it is essential to prevent water drops falling on the naphthol prepared fabric since these cause colour stains when the subsequent coupling process is carried out. Mecheels has examined the luminescence of a fabric thus water-spotted. It appears that three zones form around the water drop. The first zone shows a glaring yellow luminescence whilst the second shows a faintly dark luminescence; the third and outer zone is not luminous. On coupling this spotted fabric with a suitable diazotised base the corresponding difference in colour are revealed. Thus a naphthol prepared fabric before coupling should show a uniform yellow luminescence in ultra-violet light since it will then give a uniform dyeing. It is also found possible to distinguish between dyed goods obtained with developed direct cotton dyes and with Naphthol AS dyes since on reduction with alkaline sodium hydrosulphite these two types of dyeings then show characteristic luminescences in ultra-violet light.

Standardisation for Sulphonated Oils

A New Association formed in the United States

REPRESENTATIVES of twelve manufacturers of sulphonated oils, at a meeting in New York, have recently completed the final organisation details of the Sulphonated Oil Manufacturers' Association, a new trade body national in scope, which is comprised of most of the leading manufacturers of sulphonated oils and their products. The following officers were elected for the first year: C. P. Gulick, National Oil Products Co., president; C. M. Braham, the Arkansas Co., Inc., vice-president; H. B. Sweatt, secretary and treasurer. The offices of the Association will be at 55 West 42nd Street, New York.

This Association has been formed for the purpose of improving conditions both within the industry and between producers and consumers. To this end, a code of business practices will be adopted with the idea of promoting ethical methods of manufacturing and merchandising, to promote open and fair competition, and to discourage misrepresentations as to uses and qualities of sulphonated oils. Industry statistics relative to production, sales, shipments and stocks on hand will be compiled and circulated. Tariff and railroad rate matters also will be considered. Many oils obtained by the sulphonation process have now gained tremendous importance in the textile, leather, paper, glue and other industries; but despite this fact great confusion still exists in the various methods of analysis and nomenclatures. There are many methods of testing sulphonated oils—volumetric, rapid, cold test, separation, simplified comparative, Japan wax and ether extraction—and the results obtained from these methods frequently varies.

Aims of the New Association

It will therefore be the purpose of the Sulphonated Oil Manufacturers' Association to work with the Bureau of Standards, the American Association of Textile Chemists and Colourists, the American Leather Chemists Association and other group organisations to simplify and standardise, by nomenclature and method, the various processes used in buying and selling. It will also be one of the aims of the Association to co-operate with industries using these products; to study those problems in which the use of sulphonated oils is concerned; to discover and distribute data which will widen their uses; and to foster a closer understanding between manufacturers and users. The Association is incorporated under the laws of New York State and among the leading producers affiliated as charter members are Atlas Refinery, Arkansas Company, Inc., John Campbell & Co., Consolidated Colour & Chemical Co., A. Klipstein & Co., the Martin Dennis Co., National Oil Products Co., Onyx Oil & Chemical Co., Providence Drysalters Co., Royce Chemical Co., Salem Oil & Grease Co. and L. Sonneborn Sons, Inc.

Fastness in Relation to Leather Dyes

Effect of Metallic Salts

THE effect of the addition of metallic salts on the fastness of dyestuffs on leather is the subject of a Report recently issued to members of the Federation of Curriers, Light Leather Tanners and Dressers, Inc.

The statement has often been made that certain metallic salts, notably salts of chromium and copper, when added to the dye-bath during the dyeing process of leather have the effect of materially increasing the fastness to light of the dyed leather. This statement has probably originated by reason of the fact that in the application of certain dyestuffs to textiles, use is made of metallic salts for this purpose. The authors of this Report, M. C. Lamb, R. Denyer and J. A. Gilman, have carried out a considerable number of experiments, using a metallic salt addition to the dye-bath with the object of confirming or otherwise the statement in question, when copper acetate, copper sulphate, chrome alum, potash alum, aluminium sulphate or magnesium sulphate were employed. Experiments were first carried out by adding varying amounts of each of these salts to the dye-bath, the quantities used ranging to as large a quantity as 400 per cent. on the weight of the dyestuff—a standard acid colour. The resulting pieces of leather were then exposed to light, both direct daylight and that of an artificial sunlight lamp, but in no one case was the dyed result found to be rendered faster.

In the first range of experiments the acid dye selected was Orange II, which is a fairly fast colour to light. Experiments were subsequently repeated, using Acid Violet, which is a fugitive dyestuff, and the results obtained with the latter were confirmatory of the former, and in every case a material colour change took place even after one week's exposure to daylight.

In view of the importance of finding some means of application which would render a moderately fugitive colour faster to light, it was considered desirable to extend this work by making a number of tests, giving a pre-treatment of the leather with various metallic salts. The leather, before dyeing, was treated with various strength solutions, ranging from 0.025 to 0.3 per cent. Following upon this, leather dyed with similar dyestuffs was given an after-treatment with the salts above enumerated and in similar concentrations. The results were negative in character; in many cases the dyed results were, if anything, slightly more fugitive than when dyed without the chemical addition. Apart from this, the dyed result was, in many cases, affected in a marked degree as regards the brightness of the shade produced, the dulling being due to the production of a brown copper tannin salt resulting from the combination of the copper with the vegetable tanning agent.

Tannin in Textile Dyeing and Printing

Mordanting Properties for Basic Dyestuffs

TANNIN is placed on the market in the form of powder, needles and scales, and also in an almost chemically inert form known as "schaum." The various brands are usually graded by their actual tannin content, colour and solubility, and to a certain extent by their particular suitability for pale, medium or deep shades in the ultimate dyed effects.

Fixation on Fibre

The mechanism of the fixation of tannin is a complicated one. According to Weiss (*Textilber*, 1927, 164), it is absorbed by the cotton fibre until equilibrium is reached between the concentrations of the tannic acid in the fibre and in the liquor, but it is not known whether the subsequent fixation with antimony involves the formation of a definite compound. Optimum temperatures for the absorption and fixation have not been satisfactorily determined, but it is probable that the tannin is taken up best at about 50° C. Antimony tannate also readily forms colloidal solutions with excess of tannin, which may explain unequal dyeings and lack of fastness to rubbing and washing.

Tannin finds its greatest application in conjunction with basic dyestuffs, on account of their vividness and easy applicability in direct calico printing. In order to protect the cotton fibre in the steaming process these dyes are applied as zinc-free hydrochlorides or free bases which have to be dissolved with the aid of organic acids such as acetic, tartaric and formic. Tannin discharges are based on printing the tannin-padded article with strong alkalis, alone or in presence of alkali-stable dyes, with subsequent short steaming to remove the tannin or its antimony lake. Subsequent dyeing with suitable basic dyes then yields white or coloured patterns on a coloured ground.

The enhanced fastness of tannin-printed dyes is explained in that only on steaming does the dyestuff-tannin lake pass from the thickening to the fibre, where it is fixed. In the ordinary dyeing process an excess of antimony salt precipitates the colour lake, but apparently dissolves excess of tannin or antimony tannate. Thus in printing the tannin-dyestuff compound is formed externally to the fibre and absorbed by the latter as such.

Improving Fastness

Methods of improving the fastness of tannin dyeing or printing chiefly depend on the addition of, or "after-treatment" with, thiocarbamide, meta-phosphates, resorcinol, glycol, glycollic acid, aluminium compounds, and tungstates. An enhanced degree of wash-water and rubbing fastness is obtained, according to Ger. Pat. 371,597, by printing the basic dyes in the cold with the aid of chlorinated acetic acid or glycollic acid, or mixtures of these, or dyeing in the padding machine and fixing by drying without steaming. According to Ger. Pat. 471,788, fastness to rubbing is improved by treating the tannin-padded material with a solution of alkali stannate, silicate or aluminate, and then with aluminium acetate, formate, tartrate or sulphate. The dyeing is then carried out in an acid-free bath, to which has been added a solution of about three parts by weight of casein, two parts of ammonium borate, and about five parts of an inorganic or organic ammonium salt. The material is placed in the bath at 50° C., the temperature raised gradually to boiling and held there for half an hour, then cooled to 60° C., when the material is removed and rinsed. The proportion of casein is from 5.15 per cent. of the weight of the goods, depending on the depth of shade required.

Regarding failures, it may be mentioned that streaky effects are generally ascribed to the tannin bath being too strong, a point which often receives too little consideration in the use of this material. Care must also be taken that the tannin-padded material cannot dry off before the antimony treatment, otherwise "flocks" will be produced. The part played by tannin in calico printing with the use of sulphur colours is also of special interest, for here difficulties are due to the action of the sulphur on the copper of the printing rollers, where the formation of copper sulphide on the rollers causes wear and impairs the sharpness of the pattern. This attack on the copper can be reduced to some extent by the addition of reagents to destroy the copper sulphide, but the results are not too satisfactory.

Assaying the Quality

The quality of commercial tannins is best assayed by comparative dyeing tests. According to Heermann (*Farberei u. textilchem.* Berlin, 1929), 0.5 gramme of tannin is dissolved in hot water and the solution made up to 500 cc. and 10 grammes of sodium chloride added. Boiled cotton yarn (10 grammes) is tanned for three hours in the cooling solution, then placed without rinsing in 200 cc. of basic iron sulphate or acetate (1-2° Bé), and agitated therein for 15-20 minutes. It is then washed, and the strength of the tannin estimated from the colour. In order to ascertain the suitability of the tannin sample for pale shades, the padding is carried out as above, and fixed with an antimony salt, and the material then lightly dyed with a basic colour, such as fuchsin or methylene blue.

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.*

Deterioration of Fabrics by Dry-Cleaning

A series of tests recently conducted by the United States Bureau of Standards at the request of the dyeing and dry cleaning industry has proved that it is exposure to light and heat, rather than the chemical action of solvents which weakens the fabric. This series of tests indicated that all fabrics were weakened by repeated cleaning, but the loss of strength was in ratio to the time the materials were exposed to light and heat rather than any influence the solvents might exert. Details of the tests which were carried out will shortly be published. According to W. E. Ernley, of the Organic and Fibrous Division of the Bureau, the tests were made on materials which had been soiled by carbon black, paint, tea, syrup, gravy and lip-stick. It was found that certain solvents were more efficient on certain types of soiling in relation to the kind of material under treatment. It was also found that the amount of moisture in the material had a decided influence on the efficiency of some solvents.

Fastness of Dyes

Addressing the Society of Dyers and Colourists at University College, Nottingham, on March 2, Dr. F. W. Cunliffe, of the Wool Industries Association, Leeds, said that though much had been written on the features of coloured materials towards light, it was not until comparatively recently that the problem had been attacked in a thorough manner from all its aspects in the laboratories of the Wool Industries Research Association. The main factors which controlled the amount of fading were now fairly well established. They could be grouped under four headings: (1) the light itself; (2) the atmosphere in which the patterns were exposed; (3) the nature of the fibre or other support carrying the dye; and (4) the nature of the dyeing, such as the dyestuff concentration and methods of dyeing. Temperature also had an appreciable effect on fading, although until recently it had always been considered to be of the small account. Of atmospheric impurities, probably the most important was sulphur dioxide, and this gas might either increase or decrease the fading, or change the hue. The nature of the fibre had a very considerable influence on the fastness of some dyestuffs. It was, for example, usually found that direct colours were faster on viscose than on cotton, while some dyes were no less than five or six times as fast on wool as on cotton. With most dyestuffs on wool, the amount of fading was usually greater when the cloth was in an alkaline than in an acid state, but a few dyestuffs give the opposite result. A deep shade of a given dyestuff usually faded less than a pale shade, so that from this and the other considerations it was scarcely possible to ascribe any particular standard of fastness to a dyestuff, but only to a particular dyeing.

Leather Storage Problems

The storing of chrome-tanned leather is dealt with in the Reports of the Department of Scientific and Industrial Research for the year 1930-31. This is of special interest to leather dyers, as one of the problems of the chrome leather tanner is the storage of chrome-tanned leather in the blue—that is, after it has been tanned, but before dyeing and finishing. If the wet leather be air-dried for storage, the resulting product will not take up water easily, nor will it return to the condition in which it was before drying out. It would be of advantage to the chrome leather tanner if the leather could be dried out after tanning, as in the case of vegetable-tanned leather, and stored in the crust condition. The Leather Manufacturers' Research Association has been studying this problem, and several materials have been discovered which, when applied to the chrome blues, enable them to be dried out, stored in the dried condition, and then "wet-back" as required for finishing. Unfortunately, difficulty has been experienced in dyeing and finishing leather treated in this way, but experiments are still being carried on, and it is hoped that this difficulty will be overcome.

New I.C.I. Acid Dyestuffs

Naphthalene Orange ROS, a new addition to the I.C.I. range of acid dyestuffs, is a level dyeing acid colour giving bright reddish orange shades. It is suitable for all purposes where acid colours of good average fastness come into consideration, particularly as a basis for deep brown shades, although at the same time being suitable for shading purposes generally. It possesses good affinity for the wool fibre when dyed from a neutral dyebath, and is very suitable for use in the dyeing of union materials. On account of its excellent penetrative properties Naphthalene Orange ROS is of special value for the dyeing of wool and fur felt hat bodies, and is also of special interest for the dyeing of paper on account of its bright reddish orange shade. It possesses only moderate fastness to light, but is unaffected by the action of alum or organic acids. It is specially suitable for the production of bright reddish orange shades on jute and coir, and is also of interest for use on vegetable and chrome tanned leathers. In wool printing it is of value for use as a ground shade for the production of white discharge effects by means of the Formosul or tin discharge methods. It possesses very good affinity for the wool fibre when dyed from a sulphuric acid dyebath, and the colour is absorbed at a rapid but regular rate between 140° F. and the boil. Maximum exhaustion is obtained by raising the temperature of the dyebath to the boil and boiling for a short time in the manner usual for acid colours. Under normal circumstances this new colour is dyed from a sulphuric acid dyebath, such as one comprising Naphthalene Orange ROS 1 per cent., Glauber's salt crystals 10 per cent., and sulphuric acid 3 per cent. The material is entered and the dyebath raised to the boil, dyeing being continued for 30 minutes, when a further addition of 1–2 per cent. of sulphuric acid may be made to obtain maximum exhaustion.

Tartrazine KS

Tartrazine KS, which is also an acid dyestuff, gives bright pure yellow shades of excellent fastness to stoving and carbonising and possessing very good fastness to light. Although of all-round utility for those purposes where bright acid yellows of excellent levelling power come into consideration, it is of special value for the dyeing of carpet yarns and hosiery yarns. It possesses the important property of being unaffected by any residual alkali left in the fibre from washing treatments. This property is one that is of particular interest for the production of fast washing shades on hosiery yarns. It is specially valuable as a basis for brown or similar mode shades on yarn and piece goods, a point of interest for piece dyeing being that cotton and artificial silk effect threads are left unstained.

In view of its excellent levelling and penetrative properties, this colour is also of interest for dyeing wool and fur-felt hat bodies. It is suitable for the direct printing of wool, natural silk and tin-weighted silk, and is of value for the production of ground shades to be discharged to a white with Formosul. Tartrazine KS possesses excellent affinity for the wool fibre when dyed from a sulphuric acid dyebath. Dyeing is commenced at about 105° F. and the dyestuff is absorbed at a uniform rate as the temperature of the dyebath is raised and the maximum exhaustion is obtained at the boil.

Rhodamine 6GDN, a basic dyestuff, is of considerable importance in all branches of the textile dyeing and printing trades. A shade card issued shows sample dyeings on tannin mordanted yarns, spun silk, woollen yarn and direct tannin on cotton fabric. Rhodamine 6GDN is stated to be suitable for printing yellowish-red shades on cotton, wool, silk and rayon materials; to possess the valuable property of dyeing resistant to reducing agents; to be of importance for dyeing on discharged tannin mordants, and to be of interest for the dyeing of wool where bright shades of good fastness to stoving and milling are required.

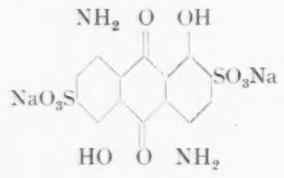
Dyestuffs Licences for February

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during February has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee. The total number of applications received during the month was 676, of which 545 were from merchants or importers. To these should be added six cases outstanding on January 30, making a total for the month of 682. These were dealt with as follows:—Granted—655 (of which 635 were dealt with within seven days of receipt); referred to British makers of similar products—20 (of which 17 were dealt with within seven days of receipt); outstanding on February 29—seven. Of the total of 682 applications received, 652 or 96 per cent. were dealt with within seven days of receipt.

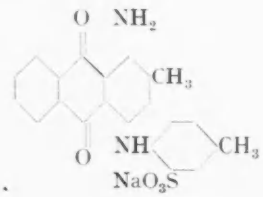
Dyes and their Application: Recent Technical Progress

By L. J. Hooley

THE acid alizarin dyes are, as is known, a development arising out of the mordant blues of the alizarin series. The connecting link between the two was Brilliant Alizarin Cyanine 3G, which was first used as a mordant colour and then found to be quite a good acid one. It is a mixture of aminohydroxyanthraquinone sulphonic acids obtained from 1 : 5-dinitroanthraquinone by heating with fuming sulphuric acid, boric acid and sulphur. The next acid colour Alizarin Saphirol B, a colour which is still extensively used, was discovered by R. E. Schmidt. This was similar in constitution to the Cyanine 3G, but a more definite product, consisting principally of 1 : 5-diamino-4 : 8-dihydroxyanthraquinone-2 : 6-disulphonic acid. Then following this came the Cyananthrols, which differed from the above in being 1 : 2 : 4-derivatives of anthraquinone.



I.
Alizarin Saphirole B.



II.
Cyananthrol RA.

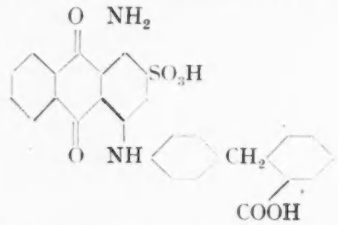
Practically the whole of the acid alizarin blues follow one or other of the two types given above, the latter claiming the higher proportion of later dyestuffs, probably because having the phenylamino group offers more scope for variation by substitution. The Alizarin Direct Blues were the next in the field, one of them being the same as I. but carrying ethylamino- instead of amino-groups and the other similar to II. but having a bromine atom in place of the methyl group, and a second sulphonic group in the 5-position of the anthraquinone nucleus. With the omission of this sulphonic group, Alizarin Sky Blue B was obtained. This along with Anthraquinone Blue SR and Erweco Acid Alizarin Blue R were the principal remaining members brought out before the war.

Anthraquinone Blue SR has a structure embodying the features of types I. and II., being 1 : 5-diamino-2 : 6-dibromo-4 : 8-disulphophenylaminoanthraquinone and Erweco Acid Alizarin Blue R being closer to this in constitution than to any of the others.

A development from the Alizarin Sky Blue B, beginning in 1913, was the series of products made by replacing a halogen atom with a sulphonic group by treating with a sulphite (generally an alkaline sulphite in aqueous phenol medium). This provided an alternative to the usual sulphonation, and was found to give products dyeing in shades with greater brightness. As an example, 1-amino-4-anilidoanthraquinone-2-sulphonic acid may be mentioned, this being made from the corresponding 2-bromo derivative. With the sulphonic group in the 2-position, an aniline group appears to be sufficient in the 4-position, instead of the *p*-toluidine one previously found necessary. Having slighter greater brightness and fastness than sky blue this product also falls behind the saphirols in levelling properties. An analogous way of making the same products was that of treating the 1-amino-4-halogen-anthraquinone-2-sulphonic acids with arylamines, in aqueous medium with a copper catalyst. (B.P. 10,378/14, Agfa.) Later in 7,861/15 (*idem.*) the same condensation was effected without the copper. These dyestuffs belong to the Direct Blue A type.

In B.P. 290,636 (*idem.*) and others we see the introduction of substituents into the arylamine nucleus, and beginning with the use of *p*-aminoalkylanilides as amines a number of similar substituted phenylenediamine derivatives have been recommended such as *m*-aminomethylacetanilide and *m*-aminobenzylacetanilide. Hydroxy substituents have also been used, thus in B.P. 360,293 (*idem.*) 1-amino-4-halogenanthraquinone-2-sulphonic acids are treated with aminophenols in weakly alkaline aqueous solution, a copper salt being present. More complicated hydroxy derivative are obtained by using aminophenol ethers of polyhydric alcohols, such as *p*-aminophenol glycol ether (D.R.R. Anm. I. 38,785, I.G.F.A.-G).

Much more complex bodies are those of B.P. 355,716 (*idem.*) where amines such as aminodiphenylmethane carboxylic acids giving dyes of constitution similar to III. are used.



With nearly all the products mentioned in the latter part of these notes, at least two general ways of synthesis are possible, the first being by reacting the amine with the 1-amino-2 : 4-dihalogenanthraquinone and then replacing the 2-halogen substituent with a sulphonic group and the second one being similar, except that the 2-sulphonic group is introduced before reacting with the arylamine.

The above only deals with the blues of this series and does not include the violets, blue-greens, greens and blue-blacks. These latter, although important, are less so than the blues, and although they are similar in constitution, the line of development does not show up as clearly as with the blues.

The Fastness of Coloured Materials to Light

By P. W. Cunliffe, Ph.D., F.I.C.

The problem of the fastness to light of coloured materials is one that has exercised the minds of dyestuff manufacturers, dyers, and the public for many years. We give below extracts from a paper read before the Society of Dyers and Colourists, at their meeting at Nottingham, March 2. The author is connected with the Wool Industries Research Association which carries on its activities at Torridon.

IN 1927 a joint committee was set up by the Wool Industries Research Association and the Society of Dyers and Colourists, to undertake work on the problem of fading, and the object of this committee was to arrange the research to be carried out on the standardising of the methods of testing the fastness of dyed materials to light, washing, and perspiration.

The chief factors which affect the amount of fading by light are: (1) the quality and intensity of the light; (2) the relative humidity, temperature and impurities of the atmosphere; (3) the type of fibre, its acidity or alkalinity, and after-treatment; and (4) the concentration of dyestuff and method of dyeing.

Direct Action of Light

Coloured materials are affected, in general, by light of all parts of the spectrum, with the exception of the red end, which appears to have little action. It is doubtful whether the small amount of ultra-violet present in sunlight has a large fading effect, but there is little doubt that the large amount of ultra-violet in the carbon and mercury arcs is very destructive. It is probable that there has to be a certain intensity of light, or threshold value, before any chemical change can take place. Ostwald, in 1922, stated that if this threshold intensity is exceeded for a brief period only, the small amount of chemical action caused by the light can be reversed in the dark. If this is so, the alternations of night and day tend to diminish fading. On the other hand, the fading produced by a continuous exposure has been compared with that of an intermittent one of equal total duration, using the carbon arc both without and with humidity control. It is found that with all the colours tried there is more fading with the intermittent than with the continuous exposure. The explanation may be due to the absorption of moisture by the patterns between the exposures, the extra moisture having the effect of an exposure in an atmosphere at a higher humidity.

Influence of the Atmosphere

In regard to the atmosphere it has been known for many years that a moist atmosphere produces an increased amount of fading. In recent years this had been put on a quantitative basis, through measurements of the colour of the surface before and after exposure at different humidities. An example of the importance of testing all dyestuffs for their reaction to humidity may be given. In 1928, a large series of fading tests were carried out in different parts of the world and it was found that whilst the majority of colours faded to a greater extent in hot places, than in Leeds, nevertheless there were some of which the reverse was true. Two cotton colours, Chlorazol Fast Pink BK and Chlorazol Fast Red K faded equally at Bangalore in India and Calistoga in California, but at Leeds the pink faded appreciably more than the red. Tests in the laboratory showed that the fading of the pink was appreciably increased by rise of humidity but the red was not seriously affected. This difference in behaviour explains why the pink was more faded than the red at Torridon, where the humidity was relatively high, but equally faded at Bangalore and Calistoga where lower humidities prevailed.

Another example of this is found in indigo, which has been used as a standard of fastness. It has been reported in the literature that a dyeing of indigo was scarcely faded in several months' exposure in a hot dry summer, but faded in a few weeks in spring. It is now known that this colour is very sensitive to humidity which, of course, was higher in the spring than in the particular hot summer mentioned.

Temperature

Until recently temperature has always been considered to have very little effect on the rate of fading, but within the last few years work carried out at Torridon has shown that it has a considerable effect. It appears probable that the earlier workers failed to keep up the relative humidity of the atmosphere surrounding the patterns, so that the tendency to produce more fading by a rise in temperature was counterbalanced by the tendency to fade less in a drier atmsophere. As an indication of the importance of this effect, it is found that at the low humidity of 30 per cent. many colours on wool fade 1½ times faster at 60° C. than at 25° C., while at saturation the factor is anything from 2 to 5 times.

The main impurity in the atmosphere of an industrial district is generally taken as sulphur dioxide though when calculated by volume the amount appears rather small. Figures given in the recently issued 17th Report of the Department of Scientific and Industrial Research on Atmospheric Pollution show that it is seldom that the amount exceeds 1 part per million and is generally much less. The sulphur dioxide present in towns may not only affect the amount of fading but may also alter the hue of the colour. Each colour, moreover, should always be tested both in the dark and in the light for its reaction to sulphur dioxide. In the dark SO_2 may darken, lighten or change the hue, while in the presence of light, it may increase or decrease the fading.

Peculiar Features of the Fibre

A given dyestuff is usually rather faster on viscose than on cotton, while some are very much faster on wool than on cotton. The nature of surface of the fibre also has its effect, a rough surface usually showing more change than a smooth one in a given time of exposure. The majority of colours on wool undergo more change when the cloth is left in the alkaline state than when in the acid state, but there are a few dyes of the type which are reduced by SO_2 of which the reverse is true. There also appears to be no doubt that the fastness of dyeings can be influenced by the addition of substances subsequent to dyeing, the effect of copper, chromium, and other salts being well known. Light shades fade more rapidly than the darker ones, using the same dyestuff. Barker, Lambert and Hirst in 1917 showed that the number of colour units lost in a given exposure is the same for different shades of the same dye, and therefore the percentage loss is greater with pale than with dark shades. At first sight this is difficult to follow, because it would be expected that the deep shades would loose more actual colour units since there is more dye present to absorb light, and therefore more likelihood of chemical change. This question, however, has been recently re-examined in detail, when a large number of dyestuffs were each being exposed for five or six different periods. Here it was found that the loss of actual dyestuff, as opposed to the colour units, was proportional to the amount of dye present, and it so happens that these proportions form an equally spaced series to the eye.

Regarding the subject of standards, if we had a perfectly constant source of light, either natural or artificial, and if we could reproduce the other conditions of exposure which have been outlined, when we wished, we could express the fastness of any colour by the number of hours required to fade. Of course, the fastness would not be quite the same under any other set of conditions, but we could generally make these the same as those existing during the use of the coloured article. Unfortunately, however, this is not quite so simple, and the best solution seems to be to adopt a set of standards against which all other colours can be judged, but it would be better to have a set of standards on the same support as that which we are testing, whether cotton, wool, paper or any other substance, since this would allow to some extent for the peculiarities of each support.

German Exports of Bleaching Compounds

DURING the first six months of 1931 exports of bleaching compounds from Germany were 14,075 metric tons compared with 13,491 tons for the same period of 1930 and 16,237 tons for 1929. During the first six months of 1931 the chief export markets were British India, 2,740 tons; Finland, 2,654; Sweden, 1,882 and the Netherlands, 1,766 tons.

Colloidal Aspects of Wool Dyeing with Acid Dyes

By Herman Boxser

In this article, reprinted from a recent issue of the "American Dyestuff Reporter," the author discusses the application of acid dyes to wool from a colloidal standpoint, especially as regards the action of acid dyes which exhibit neutral dyeing properties.

THE resemblance of the structure of wool to those of colloidal gels has been emphasised by J. B. Speakman (*Jour. Text. Inst.*, 1926, *17*, T457). It had been the opinion of previous investigators that the wool fibre possessed a two phase gel structure, namely, a fibrillar structure which enclosed a viscous medium, one in physical equilibrium with the other. Speakman, however, found that wool fibre consisted of two gels arranged in parallel. The first gel might be called a petrified gel and comprises an elastic cell wall which encloses a fibrillar structure which is *not* in physical equilibrium with a viscous phase. The second gel fills the interstices of the above mentioned petrified gel and is capable of reversible solution in, or deposition from water.

Micelle Structure of Wool

Most recent researches tend to emphasise the micelle structure of wool. At present when cellulose is mentioned, we visualise this fibre as composed of micelles or large crystallites to which cellulose owes its chemical and dyeing properties. If a micelle structure of wool is clearly demonstrated we may then regard the wool fibre as somewhat analogous to cellulose in regards to the adsorption theory of dyeing. Asbury (*Jour. Text. Science*, 1931, *1*, 28) in executing X-ray investigations of the inner structures of wool found a distinct crystalline pattern, stating that the elements of wool fibre are shown to be built up in a regular 3-dimensional pattern. The crystals are minute and their aggregation is colloidal. X-ray photographs showed that stretched and unstretched wool have different crystallographic structures, the difference being ascribed to the possible presence of a beta and an alpha keratin.

Instead of being a simple molecular body to which a definite formula might be given, wool is composed of several chemically distinct substances. The principal component of the fibre is a protein, keratin, whose composition is approximately as follows:—Carbon 50 per cent., hydrogen 7 per cent., oxygen 26-22 per cent., nitrogen 15-17 per cent., sulphur 2-4 per cent. This compound differs from other proteins in the large proportion of sulphur present, much of which appears to be in a state of loose combination. S. R. and E. R. Trotman and R. W. Sutton (*Jour. Soc. Chem. Ind.*, 1926, *45*. 30) claim that sulphur, however, is not a structural constituent of wool fibre.

Theory of Acid Dyeing

Acid dyes are sodium or calcium salts of sulphonic acid derivatives. They are applied to wool either from an acid or neutral bath, the manner of application depending on the properties of the respective dyestuffs. The function of the acid is to liberate the free colour-acid from the dyestuff, which combines with the fibre-base. The principal assistant employed in acid dyeing is Glauber's salt, which acts as a retarding agent. As Glauber's salt is a product of the interreaction between the dye salt and acid, its excess presence will retard this reaction, since it is a principle of chemistry that when one of the products of a chemical reaction is present in the solution, the rapidity and extent of the reaction will be reduced. Here then we have a purely chemical explanation for the action of acid dyes on wool. The colloid chemist, however, has advanced a theory which regards the acid dyeing of wool as a colloidal phenomenon, depending on the theory of adsorption. This has been conceived by W. D. Bancroft (*Jour. of Phys. Chem.* 1914, *18*, 2) who denies the existence of a chemical reaction between the wool fibre and the dye.

The addition of a strongly adsorbed cation will increase the amount of dye taken up, other factors remaining constant. This occurs when upon the addition of the hydrogen ion of an acid, greater absorption of dye is effected. In practical dyeing, neutral dyeing acid dyes are applied to the greatest extent in cotton-wool union fabrics by the one-bath process, in which the wool is dyed by this class, and the cotton coloured by those substantive dyes which possess little affinity for wool even at the boil. In this operation, the practical dyer disregards theory, when in the event that the shade of his wool is weak, an addition of Glauber's salt is often made to throw more of the neutral dyeing acid dye from the solution on to the fibre. The effect of this procedure can be check, if two 2 per cent. dyeings are made on wool of Alphazurine A which is a neutral dyeing acid dye of the Patent Blue family; one dyeing containing no Glauber's salt and the other 20 per cent. of Glauber's salt. It will be noted that the wool dyed in the bath containing Glauber's salt, possesses a much heavier shade than that dyed with the dye alone. Glauber's salt here acts not as a retarding agent, but as an exhausting agent instead.

Behaviour in Neutral Solution

In working with certain acid dyes on wool which possessed good fastness properties in all respects, the author was struck by the unusual action of these dyes in behaving not unlike substantive dyes. These dyes possessed great affinity for wool from acid dyebaths, so much in fact, that the weakest exhausting assistant had to be employed for level results, *i.e.*, ammonium acetate. Their similarity to substantive dyes existed in their property to assume a turbid condition soon after 1 per cent. stock solutions were prepared, a characteristic displayed by typical substantive dyes like Benzopurpurine 4B. This would tend to indicate a condition of greater molecular aggregation at lower temperatures, a property of many colloidal dye sols. The author therefore decided to select one of these dyes, Polar Orange R, and note its behaviour with wool from neutral solution. A 2 per cent. dyeing for 30 minutes at a boil showed a marked affinity of the dye for the fibre, and a similar dyeing with the addition of 20 per cent. Glauber's salt produced a much heavier dyeing. This was a characteristic of a typical neutral dyeing acid dye.

In experimenting with basic dye solutions (*Melliand*, 1931, *2*, 11) it has been found possible to reverse one of these dyes from true to colloidal solution, and in this manner were successful in dyeing viscose directly, without a mordant, with fair fastness. A 2 per cent. dyeing of the Polar Orange R with the addition of 20 per cent. Glauber's salt was therefore made on cotton, and the affinity of the dye for the fibre noted. There was a marked affinity with excellent exhaustion of the dyebath. The dyed fibre exhibited fair fastness when subjected to a washing test, the fastness in this respect being comparable to certain substantive dyes. A neutral dyeing of the acid dye was then made with the addition of Glauber's salt to a piece of cotton-wool union goods for the purpose of comparing the relative affinities of the dye for cotton and wool from a neutral bath. A fairly good union dyeing was produced, the wool being dyed slightly heavier than the cotton.

The best explanation for the behaviour of neutral dyeing acid dyestuffs is that contained in a study of the physical chemistry of dyeing by Briggs and Bull (*Jour. Phys. Chem.*, 1922, *26*, 845). The conclusions drawn by these investigators are: (1) The adsorption of acid dyes by wool is a strictly adsorption phenomenon; (2) all dyes behave like substantive dyes if their solution becomes colloidal (for this reason, Methylene Blue, as soon as it has been rendered colloidal acts like a substantive dye and no longer behaves as a true basic colour); and (3) the restraining effect of sodium sulphate on acid dyes may pass through a maximum as the sulphate concentration is increased. Coincidental with these conclusions, the author is of the opinion that many acid dyes in neutral solution exhibit colloidal properties, and in this state behave much like substantive dyes. The addition of an electrolyte, like Glauber's salt, causes a destabilisation of the dye solution and causes a greater exhaustion of the dye. It appears, however, that the molecular condition of these dyes is such that the dye aggregates may be of smaller size than that of substantive dyes.

The World Dye Situation Reviewed

A Survey of Statistics from the Principal Producing Countries

The following notes are abstracted from a lengthy survey on the dye situation in the principal producing countries, compiled by the United States Department of Commerce and published in a recent issue of "Commerce Reports."

IN the major European dye-producing countries there is now a concentration of manufacture control in the hands of national groups. Participating manufacturers claimed satisfactory operation of the Franco-German-Swiss dye sales agreement during 1930 and 1931; but in this limited period and with present disturbed world conditions, it is difficult to kauge accurately the intensity of international competition. Outstanding events in the German dye industry during 1931 were the acquisition by the I. G. Farbenindustrie of an interest in the Italian dye industry and an improved condition in German foreign dye markets. The third quarterly report of the I. G. indicated that this favourable condition has been affected somewhat by currency depreciation in certain European countries but offset by seasonal increases in oversea trade. The German Dye Trust has practically completed its 5-year programme of rationalisation and concentration and increasing efficiency through more extensive machine operation.

Switzerland and France

During the first 10 months of 1931 conditions in the Swiss dye industry showed a slight improvement. The Franco-German-Swiss dye sales agreement and the Basle I. G. are reported to have been important factors in keeping sales at present levels. Recent Ciba statutory changes have enhanced the possibility of strengthening the Basle I. G. through the formation of a single holding company. It is estimated that 90 per cent. of the Swiss dye output is exported, on which basis the 1930 production is placed at 20,039,000 lb. On this basis it is believed that the 1931 output during the first 10 months is slightly above the 1930 level. Speciality dyes have continued to receive special attention. The lower priced dyes are produced principally by branch factories of Swiss chemical firms in foreign countries.

France has also continued to progress in the field of finished dyes. The industry was slightly below normal during the greater part of 1930 but during 1931 the trade has improved considerably. In 1931 French production supplied 96 per cent. of the apparent domestic consumption. The Etablissements Kuhlmann continue to control directly about 70 per cent. of the entire French production, while the Société des Matières Colorantes et Produits Chimiques de Saint Denis, with whom the Kuhlmann interests have a trade agreement, provides approximately 24 per cent.

Japan, Italy and Russia

The manufacture of several new dyes has been undertaken in Japan, supplementing the range of colours produced. It is generally anticipated that the distinct geographical advantage of the Japanese dye industry in the markets of the Far East will be somewhat offset as a result of recent political and economic disturbances in these countries. Although Japan ranks only seventh on the basis of quantity dye output, it is an important factor in the international dye trade, due largely to Government fostering and subsidy, as well as its advantages geographical proximity to the important dye markets of the Far East. In 1930 there were 19 factories producing coal-tar dyes, of which 3 produced intermediates and 14 both intermediates and dyes. Domestic production supplied 68 per cent. of the consumption in 1929 and 79 per cent. in 1930.

The principal recent development in Italy has been the reorganisation of the Aziende Chimische Nazionale Associate A.C.N.A.) of the former Italgas group under the name "Aziendi Colori Nazionale Affini." The A.C.N.A. is capitalised at 60,000,000 lire, held jointly by the German I. G. Farbenindustrie and the Italian Montecatini, in the proportion of 29,000,000 and 31,000,000 lire, respectively, and will continue to operate the dye plants of the former A.C.N.A.

A comparatively recent development of importance to the world dye trade has been the rise of Russia as a major producer, coincident with its "chemicalisation" programme under the 5-year plan. Russian dyes, however, are being consumed almost entirely in the home market; imports are still sizable and the effect of the growing production has not been felt greatly in international export trade. No official figures are available for the output of coal-tar dyes in Russia. Production of dyes in the fiscal year ended September, 1930, as compared with 1928-29 figures, in parenthesis, as follows: Sulphur black, paste, 22,309,000 lb. (18,455,000); other sulphur, dry, 1,023,000 (877,000); direct cotton, 6,455,000 (4,250,000); acid, 3,135,000 (1,755,000); basic 362,000 (357,000); mordant, 256,000 (353,000); fur, 167,000 (108,000); nigrosine, 1,001,000 (738,000); other, 280,000 (935,000); total 35,078,000 (27,873,000). Unofficial estimates for the 1930-31 output indicate that it may be still larger.

Increased German Output

In the absence of official production figures, the German exports generally are estimated to represent 60 to 70 per cent. of the total output. On this basis an improved condition in the German dye output is indicated by increased exports during 1931. The German output in 1930 was reported to have supplied about 90 per cent. of the apparent home consumption. The I. G. Farbenindustrie usually is credited with controlling the entire output. Domestic production supplied 85 per cent. of the apparent British consumption of dyes in 1930. Previously, Imperial Chemical Industries manufactured about half of the entire British production, and with the acquisition of the British Alizarine Co. it may be expected to have supplied a still larger proportion of the output in 1931.

World Exports Decline

Total exports from the seven leading world coal-tar dye exporting countries in 1930—Germany, Switzerland, the United States, Great Britain, France, Japan and Italy—showed an aggregate decline of 17,273,700 lb. and valued at £798,000, or 9.7 per cent. by quantity and 5.2 per cent. by value from the 1929 level, emphasising the universal trend toward the more expensive fast dyes and colours of greater concentration. Of these exporters, Italy and Japan alone recorded increases in 1930. During the first half of 1931 exports from Germany, Japan and France alone were above the level of those of the corresponding period of 1930. Since then a more general improvement has been noted, and available figures for each country during 1931 showed marked increases above those of comparative periods in the previous year, except in the case of the United States and Italy.

Exports of coal-tar dyes, colours and stains from the United States during the first 10 months of 1931 amounted to 17,840,000 lb., worth £820,000, compared with 22,648,700 lb., valued at £1,020,000, during the corresponding period of 1930. The chief countries of destination during the first 10 months of 1931 follow: China, 11,011,400 lb., £350,000; Japan, 1,232,700 lb., £140,000; Canada, 1,410,100 lb., £130,000; British India, 840,200 lb., £70,000; Belgium, 368,600 lb., £18,000; Germany, 462,300 lb., £18,000.

Official statistics of the German dye trade do not present a complete picture of its international activities. German interests own or control plants in a number of foreign countries which at times also handle products not of their own manufacture. Exports during the first nine months of 1931 amounted to 74,128, 900 lb., compared with 69,471,600 in the corresponding 1930 period. During the first nine months exports were distributed as follows: China, 30,321,200 lb.; British India, 9,979,200 lb.; Czecho-slovakia, 4,017,000 lb.; Great Britain, 2,886,000 lb.; Japan, 2,466,700 lb.; the Netherlands, 2,456,200 lb.; the United States, 2,088,200 lb.; Belgium, 2,066,100 lb.; other countries, 17,848,000 lb. These eight countries absorbed 75 per cent. of the entire German exports dutink 1931.

Export Statistics and Destination

During the first half the 1931 the export decline in Switzerland was marked. Since then shipments have risen rapidly,

and at the end of October, 1931, amounted to 15,565,900 lb., exceeding the 15,411,600 lb. exported in the corresponding 1930 period. During the 1931 10-month period the following were the chief countries of destination: China, 2,827,300 lb.; Germany, 2,687,000 lb.; Great Britain, 1,451,400 lb.; the United States, 1,141,900 lb.; France, 888,700 lb.; Czechoslovakia, 808,300 lb.; Japan, 792,300 lb.; Italy, 708,600 lb. Belgium, 622,100 lb.; British India, 431,400 lb.; other countries, 3,206,600 lb. These 10 markets absorbed 79 per cent. of the total shipments during 1931.

Total exports of British coal-tar dyes during the first 10 months of 1931 increased to 11,041,400 lb., from 10,388,800 lb. in the corresponding period of 1930. In 1930 British India was the best market for British alizarine and anthracene dyes; China and Japan for synthetic indigo; and China, Australia, Spain, Belgium and France for other synthetic dyes. During 1931 French trade has shown improvement, exports during the first eight months increasing to 3,898,400 lb., from 2,717,200 lb. in the 1930 period. It is believed that Belgium-Luxemburg, Japan, China, Germany, Great Britain, Italy, the Netherlands, French Indo-China and Spain were the chief countries of destination. Japan, despite generally adverse trade conditions, increased its exports more than 150 per cent., to 4,598,700lb., worth £80,000, in 1930, of which 4,305,300 lb. went to China. The increase continued in 1931 at a less rapid rate, exports during the first nine months, 3,895,600 lb., being 75 per cent. above the figure for the corresponding period of 1930. Italian exports of synthetic dyes registered an increase to 1,166,200 lb. in the first eight months of 1930, but fell to 950,800 lb. in the 1931 period. No destination data are available, but it is reported that dyes go to Germany, France, the United States, Great Britain, Switzerland, British India, China, Japan, Spain, Austria, Argentina and Brazil.

Imports of Producing Countries

Imports into Germany, Switzerland, France and Italy during 1931 have declined below the level of corresponding 1930 periods, accompanied in the case of the first three by increased exports. Imports into the United States, Japan and Great Britain, on the other hand, have increased. General imports into the United States of coal-tar colours, dyes and stains, colour acids and colour bases, m.e.s., during the first 10 months of 1931 amounted to 4,247,000 lb., valued at £950,000, a slight increase over the 4,187,400 lb., invoiced at £890,000, received during the 1930 10-month period. Germany's total imports of coal-tar dyes declined markedly during the first nine months of 1931, incoming shipments amounting to 4,540,900 lb., compared with 5,254,700 lb. in the 1930 three-quarter period. Switzerland, the Netherlands, and the United States were the major sources. Imports into France during the first eight months of 1931 decreased sharply to 1,642,400 lb., from 2,134,500 lb. in the 1930 period. Imports into Switzerland during the first 10 months of 1931 declined to 1,470,000 lb., from 1,714,100 lb. in the corresponding 1930 period. Germany and France are generally the leading suppliers of dyestuffs to Switzerland.

Although British imports of coal-tar during the first 10 months of 1931, amounting to 4,601,800 lb., still were above the 4,481,200 lb. received during the 1930 period, indications are that the currency depreciation has somewhat reduced incoming shipments. This partially offsets the stimulation given imports during the year by changes in the dyestuff import regulation act in respect to import license. Germany and Switzerland are generally the leading countries of origin for British dye imports, while the vat and the direct cotton colours form the chief type of synthetic dye imported.

Dyes for Hemp and Straw

A COLOUR card showing recommendations for dyeing the standard shades of the London and Luton Bleachers' and Dyers' Association for Spring, 1932, has been issued by Imperial Chemical Industries, Ltd. In all fifty-two colours are given, the lighter shades being produced on bleached hemp. For hemp the dyestuffs used are exclusively those in the Chlorazol range; for straw Coomassie colours are used in combination with Naphthalene Reds and Naphthalene Browns.

Imports of Dyestuffs in Yugoslavia

THE dye requirements of Yugoslav cotton and woollen mills are supplied almost entirely from foreign sources since there is very little production within the kingdom of either natural or synthetic dyes. The imports of dyes have increased gradually, attaining a value of $1,202,000 in 1930. Germany is the largest source, Switzerland and Czechoslovakia practically supply the remainder of the demand. Fair quantities of natural dyes are also consumed by the rapidly expanding textile industry, particularly in the knitting and cotton branches. In 1930 imports of natural dyes were valued at $20,000. France and Greece furnished practically the entire demand.

Scottish Dyestuffs Market

THE Scottish woollen manufacturers had a very successful joint exhibit at Olympia, the exhibit being organised by the National Association of the Scottish Woollen Manufacturers. This is the first time that both large and small manufacturers have combined for an exhibit of this character, and the step is indicative of the common feeling of the manufacturers that they must get together for the good of the trade.

Although only a few of the tweed manufacturers can be said to be doing a fair amount of business, orders are tending to improve, more repeat orders having come in. It is considered that although the tariffs will ultimately be an advantage, that the trade will be slow in getting well under way, as merchants will have to first recover their financial positions. Dyers are moderately well employed. It is stated that a new laundry and dyeing works is to be opened in Stirlingshire at Larbert. Recently, before the Dean of Guild Court, Imperial Chemical Industries, Ltd., obtained sanction for new recreation buildings for their dyeworks at Grangemouth.

Revival of 18th Century Shades

IN commemoration of the two hundredth anniversary of the birth of George Washington, the United States Textile Colour Card Association has just issued a special series of colonial colours, in co-operation with the United States George Washington Bicentennial Commission. These colours have been reproduced from original costumes worn by George and Martha Washington and other eminent personages in early American history. They have been taken from the famous costume collection on exhibition in the United States National Museum and are authentic and historically correct. The Association has presented these shades in the form of an historical brochure, thereby following an entirely different procedure from that regularly employed in issuing its colour cards.

"Daily Mail" Ideal Homes Exhibition

THE greatest telephone exhibit ever shown to the public will be presented by the Post Office at the *Daily Mail* Ideal Home Exhibition which opens at Olympia on April 5. A special power station will be erected and each instrument and piece of equipment shown will be laid down with a thoroughness not less than that demanded by permanent installation. The first telephone and the most modern instrument will be shown, and secrets of the world-famous Post Office engineering research station at Dollis Hill will be revealed in detail. The public will be at liberty, almost within any limits, to work the machines themselves. Friends a few feet apart will be able to talk to each other across 1,000 miles of actual underground cable. The City of Light, which will form the setting for the exhibition this year will abound with features entertaining and instructive to the home-lover. A Tudor hamlet grouped round an old-time village green and a quaint Old English Street will make an appeal no less universal than the wonderful "Gardens of the Novelists." "Y Bwthyn Bach," the wonderful little cottage which was specially built and furnished to be a gift from the Welsh nation to Princess Elizabeth on her sixth birthday in April, will be on view in its gay miniature garden. In the Theatre of Fashion will be displayed the most up-to-date creations of the most famous designers. This year the exhibition will remain open from April 5 to 30.

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.*

Textile Institute Annual Conference

Arrangements in connection with the Textile Institute Conference which is to take place at Leamington Spa in the latter part of Whit-week (May 19-20) are proceeding satisfactorily, and the prospects are favourable to a good attendance of members. The excursion part of the programme will be limited to one afternoon, when Coventry will be visited, including the works of J. & J. Castle, Ltd., and the Quinton Hosiery Co., Ltd. With the exception of the Mather Lecture, the papers to be contributed will deal with various aspects of textile testing. On the chemical side, the detection and estimation of chemical damage in wool will be dealt with by Professor P. Krais, of the Deutsches Forschungsinstitut für Textilindustrie, Dresden; chemical tests in the wool industries will be the subject of a paper by Mr. E. Hill, of the Wool Industries Research Association: and the testing of laundered fabrics will be discussed by Dr. R. E. V. Hampson, of the British Launderers' Research Association. The Mather Lecture will be delivered by Sir Michael Sadler, of University College, Oxford, who is taking for his subject "Liberal Education and Modern Business."

Textile Mill Water Supply.

The subject of *pH* control for the water supply of textile mills has been discussed in a paper read at a recent meeting of the American Association of Textile Chemists and Colourists. Every water supply and coagulating chemical has an optimum coagulation *pH* range, due to variation in colour, turbidity, natural alkalinity, and other seasonal conditions. By determining the optimum *pH* value of a water for proper coagulation, which can be done with a few simple tests, the smallest amount of coagulant necessary, to give the quickest and best "floc," is used at all times. In some plants this *pH* control has reduced the amount of alum to almost half that originally used. An overdose of coagulant is just as harmful as an underdose; by using the *pH* control, it is therefore possible to eliminate any solvent action on the "floc," which would result in the aluminium salts going on through the filters and causing considerable trouble in the various processes in the textile plant. Coagulation at the optimum *pH* value also insures higher bacterial efficiency, makes possible a decrease in size of the sediment basin and speeds up the process generally.

A New Crystal Gum for Textile Printing

The presence in a gum thickening agent of very fine, almost colloidal, yet incompletely soluble deposits, which are not removed by straining, is a frequent cause of defects in printing. But this trouble is claimed to be overcome in a new crystal gum introduced by W. A. Scholten's Chemical Works, of Groningen, Holland. Printing colours prepared with Nafka crystal gum thickening are characterised by uniformity and smoothness; they are of special value for heavy blotches; lines are sharp and clear, and cloudy colours and "blebbing" are avoided. As the colours are brighter than with other thickening agents, it is suitable for delicate and sensitive colours—*e.g.*, alizarines. Nafka crystal gum dissolves entirely in cold water in a few hours. It can be washed out much more easily than gum tragacanth and all starch products; the printed portions of the cloth are therefore quite soft and supple after the washing process. At present three brands are available; KI is claimed to be the most economical in use, in consequence of its high thickening power. A-Extra is an extremely pale-coloured gum, and is recommended for light and delicate shades; it is also somewhat better than KI for block printing. Nafka crystal gum A is especially suitable for silk printing.

Fading of Dyestuffs on Leather

Tests on the fastness to light of a number of dyestuffs on leather have been made by Woodroffe, who used gambier mordanted chrome leather in order to avoid any misleading observations due to fading of the leather itself, as might be the case with vegetable-tanned leather (*J. Int. Soc. Leather Tr. Chem.*, 1931, p. 593). The exposures were made during June to August. His conclusions are that (1) basic dyestuffs, except auramine, safranine, rhodamine and methyl violet fade rather badly; (2) with the exception of Kiton fast violet, the acid colours are fairly permanent; and (3) the direct colours are the most permanent. Exposed to daylight in vacuo, samples dyed with Bismark brown and chrysoidine faded completely, while other colours were satisfactory. The fading of dyestuffs on catechol-tanned leather is greater in atmospheres of high humidity than those exposed to a lower degree of humidity. The fading of dyestuffs on chrome leather has also been investigated by Lamb and Gilman in a report issued to the Federation of Curriers, Light Leather Tanners and Dressers Inc., using a number of acid and basic colours. Violet colours were found to be very fugitive, and browns comparatively fast to light. In all cases, the rate of fading was slower than on vegetable-tanned leather. These same authors, in a further Report, describe some tests on the effect of the addition of metallic salts to the dyebath in the dyeing of leather, it having been stated that certain metallic salts improve the fastness to light. Experiments were made using copper acetate, copper sulphate, chrome alum, potash alum, alumina sulphate, and magnesium sulphate in amounts up to 400 per cent. of the weight of dyestuff, in conjunction with Orange II and Acid Violet. The leathers were then tested by exposure to both daylight and an artificial sunlight lamp, but no increase in fastness was observed. Further tests were made, applying the metallic treatment prior to dyeing, and again afterwards, but no improvement was effected. There was, in fact, an opposite tendency coupled with a dulling in shade due to the formation of the metallic tannate during mordanting.

Dyestuffs Licences for March.

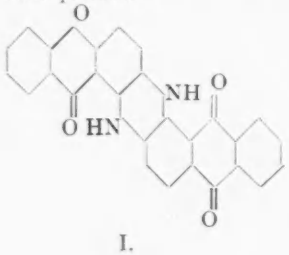
The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during March has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee. The total number of applications received during the month was 414, of which 355 were from merchants or importers. To these should be added seven cases outstanding on February 29, making a total for the month of 421. These were dealt with as follows:—Granted—400 (of which 389 were dealt with within seven days of receipt); referred to British makers of similar products—20 (of which 19 were dealt with within seven days of receipt); outstanding on March 31—1. Of the total of 421 applications received, 408, or 97 per cent., were dealt with within seven days of receipt.

Dyes and Dyeing: Recent Technical Progress

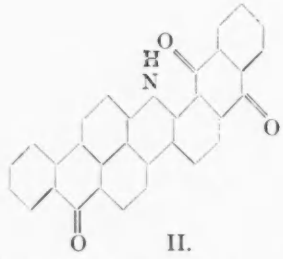
By L. J. Hooley

IT is rather remarkable that the majority of the anthraquinone vat dyes are symmetrical in structure. Thus, indanthrone, the first to be discovered, is given a formula which can be divided into two equal halves (I).

The same applies to flavanthrone, the next member to be discovered, and also to dibenzanthrone, isodibenzanthrone and pyranthrone. As indanthrone and dibenzanthrone are obtained by an alkali fusion and flavanthrone by a condensation using antimony pentachloride, the symmetrical structure may be regarded as accidental rather than designed. Pyranthrone, however, is synthesised from an intermediate, 2. 2'-dimethyl-1 : 1'-dianthraquinonyl, which is already symmetrical and which may be expected to yield an analogous final product.



I.

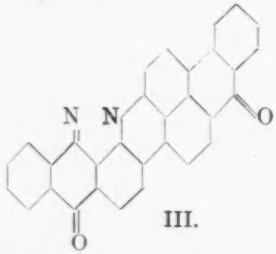


II.

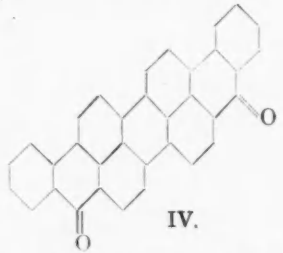
This symmetrical character is also to be seen in a large number of subsequent anthraquinone vat colours. Even if we include members such as the benzoylaminoanthraquinones and the di- and tri-anthrimides, which do not consist of a single polycyclic nucleus, we are able to make the same generalisation. As instances, we may mention Algole Red 5G which is 1 : 4-dibenzoyldiaminoanthraquinone and Indanthrene Red G, which is obtained by condensing one molecule of 2 : 6-dichloroanthraquinone with two of 1-aminoanthraquinone. More recently Caledon Jade Green has provided another outstanding example, and we have also had the dyestuffs of the anthanthrone, dipyrazolanthrone and dibenzpyrenequinone series. Among important exceptions, there may be mentioned Caledon Brown R and Caledon Olive G, although even these have partially symmetrical molecules.

Unsymmetrical Polycyclic Vat Dyes

Comparatively recently there have been synthesised a number of new complex polycyclic vat dyes, the structure of which has been characterised by the presence of two unequal halves derived from different known symmetrical vat dyes. The first one which may be mentioned stands rather distinct from the others as regards time, being discovered in 1908. It may however be mentioned as being the first. This was a green vat dye mentioned in G.P. 212,471 (B.A.S.F.) as obtained by the alkali-fusion of the benzanthronylaminoanthraquinone from Bzl-halogenobenzanthrone and 1-aminothraquinone. (See formula II.)



III.

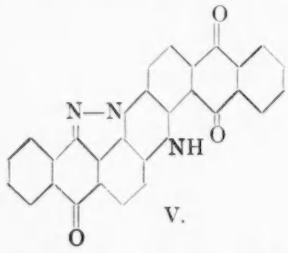


IV.

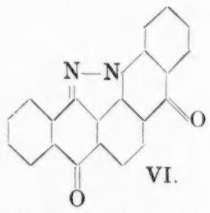
Coming now to the members described in B.P. 298,775 (I.G.), a number of pyrazolanthrone derivatives were synthesised, these being obtained by condensing halogen aromatic compounds with pyrazolanthrones in which the hydrogen of the NH group had been replaced by metal. Among the products obtained in this way, there may be mentioned that from pyrazolanthrone itself and Bzl-bromobenzanthrone, this being a yellow substance, analogous in constitution to Bzl : Bzy'-dibenzanthronyl but apparently not a vat dyestuff itself. On alkali-fusion it is converted to a dyestuff which appears to have the constitution shown in formula III. Comparison of this with that of dibenzanthrone (IV) shows the great similarity. This similarity extends to properties, the product dyeing in blue shades and being readily converted to other vat dyestuffs by halogenation and oxidation. Under more violent conditions of alkali fusion a different product is obtained.

Mixed Pyrazolanthrone Derivatives

Rather similar to the previously mentioned compound, which may be regarded as half dibenzanthrone and half dipyrazolanthrone, is the body of idanthrone-like character (V). The method of synthesising this follows similar lines to the benzanthronylpyrazolanthrone. If instead of combining the pyrazolanthrone with a simple halogen derivative it can be treated with *o*-aminohalogenoanthraquinone, *e.g.*, 1-amino-2-chloroanthraquinone, condensation appears to take place both through the NH of the pyrazolanthrone and the adjacent ortho position. Comparing the new body with idanthrone, it is seen to dye in blue-violet shades as distinct from blue. It is stated to have good fastness to chlorine and is presumably better in this respect than indanthrone, perhaps because there is only the one NH group to be attacked, B.P. 321,585 (*idem*). Still another type of mixed pyrazolanthrone derivative is the one which is half pyrazolanthrone and half acridone. In B.P. 332,316 (*idem*) 1-halogenoanthraequinone-2-carboxylic acid is condensed with hydrazine to give a pyrazolanthrone-2-carboxylic acid. This carboxylic acid is then converted to the acid chloride, condensed with a hydrocarbon such as benzene, and then cyclised to the dyestuff of formula VI. The shade appears to be substantially the same as that of the corresponding acridone.



V.



VI.

Reverting to the anthraquinonylaminobenzanthrones, further dyestuffs of this series have been made more recently. In B.P. 339,396 (*idem*) amino derivatives having similar constitution to II are converted to new vat dyes by benzoylation. It has also been found that anthraquinonylaminobenzanthrones, which contain several anthraquinonylamino radicles in the molecule, and of which one is in the Bzl'- position, can be converted to valuable vat dyes by alkaline condensing agents, and that the resulting products may be improved by a subsequent acid condensation. As an example di (l'anthraquinonyl)-6 : Bzl-diaminobenzanthrone gives an olive brown dye, which on heating in concentrated sulphuric acid is converted to a green (B.P. 337,741, *idem*).

Extended Use of Trichlorethylene

THE use of trichlorethylene in the dry cleaning industry has been increased rapidly during the last few months, owing to the introduction of a new type of machine for the individual cleaning of garments in a few minutes, as distinct from the more normal practice of putting 30 or 40 of them in a rotary washing machine for over an hour. The apparatus combines a small rotary washer with a store tank for the solvent above it, and a still below, from which clean solvent is returned to the storage tank. Other machines are using mixtures of carbon tetrachloride and benzene, and, where large enough, are combined with a solvent recovery plant. There seems little doubt that the trichlorethylene individual method will be considerably extended, and it is reported that already one-half of the "tri" sold in this country is used for that purpose and for other degreasing operations.

Dyeing and Finishing Hosiery

By W. I. Pickings

We give below extracts from a paper read before the American Association of Textile Chemists and Colourists, which was printed in full in our contemporary "The American Dyestuffs Reporter." The author is associated with E. I. du Pont de Nemours and Co.

SOME time ago, for hosiery dyeing, I would have recommended the two-bath method, but more recently one has realised some of the advantages of the one-bath method, and with some few exceptions, the one-bath method would be preferred. In discussing this matter, it would be well to outline the advantages claimed for each method. The claims of the two-bath method are economy of dyestuffs and more uniformly working formula. The claims of the one-bath method are increased production, less handling and consequently less seconds and a better finish.

It is true that the two-bath method will probably save on the cost of dyes, but I really believe that this will be more than offset by the time saved on the one-bath method. I am sorry that I have not any definite figures to give on this, but I have never had the opportunity to make any definite comparisons along this line. However, it is my opinion that taking everything into consideration the difference in the cost per dozen would be so small as to be negligible, and even then would probably be in favour of the one-bath method.

Two-Bath Process Favoured

The question of uniformly working formula, of securing check dyeings from run to run, is one which I believe influences a number of dyers to favour the two-bath method. The proper selection of colours will give just as uniform checks from run to run, in the one-bath method as in the two-bath. I do not believe that there are many silk hosiery dyers, if there are any, who do not have to make small shading additions to the majority of their runs, whether they be the one-bath or the two-bath method. The additions are generally very small, and in either case do not materially delay the completion of the dyeing. However, one point must be emphasised, and that is the selection of the proper type of dyes. There are a limited number of colours which perform well-in the one-bath method, and it is essential that these types be selected for this work. If, for any reason, these types are not available, then probably the two-bath method would be better. Some dyers claim that as a rule the one-bath method will check somewhat better than the two-bath, due to the fact that when the degumming oil or soap is put in the bath a more or less definite *pH* is established, which is varied less by changing water conditions than when just the raw water is used as a dye bath.

The matter of the finish is one which I am not prepared to discuss further than to say that in most cases a scroop or hand may be given the goods, regardless of the method of dyeing. It is claimed by some dyers that the two-bath takes some of the life out of the silk, due to removing practically all of the gum in the first bath, and then giving it 45 minutes to one hour in another boiling bath. To overcome this, some dyers have resorted to what I call a combination of both methods, which means that they partially degum in one bath, and then drop that bath and finish degumming in the dye bath, which has practically the same properties as the one-bath method.

Rayon Hosiery Problems

As I have previously mentioned, there is made in this section a quantity of ladies' rayon and mercerised cotton hose. There are several outstanding problems encountered by the dye-house man in processing these numbers. I presume one of the most outstanding problems is in the case of rayon when hard water is encountered. The combination of rayon coning oil, soap and hard water is one that has caused a lot of trouble. Some dyers prefer to adopt the two-bath method to remove the oil before dyeing. In this case, it would be best to give the material a good boiling off, starting at a point near the boil, with plenty of good soap or scouring oil. After some 15 or 20 minutes, the machine should be flushed over to prevent the coning oil and soap curds precipitated by the addition of cold water, from settling on the goods. Of course, if the mill has a hot water supply, then this would be used to advantage at this point. When the oil is thus removed, care must still be taken regarding hard water curds, but it has been my experience that these curds do not give nearly as much trouble when the coning coil has previously been removed.

Some plants find that they do not have to resort to this method, but dye by the one-bath method, and flush the machine over at the end of the dyeing period, rather than drain it. This flushing serves as a rinse, and the goods are removed without further rinsing, as soon as they are cool enough to handle. In some cases, I have found that this method gives trouble, and that it has been necessary to remove the material from the dye-bath without rinsing or cooling. In this case the goods are always sacked, and a smooth stick is used to lift the sacks from the machine. The addition of trisodium phosphate or soda ash will help in keeping out these hot water curds. There are also on the market certain scouring oils which will emulsify coning oils in hard water without precipitation of curds. Of course, the ideal solution of this problem is soft water.

The question of obtaining a good union between rayon and mercerised cotton toes and heels might also be raised. There are just two things to be considered in the solution of this problem; one is the proper selection of dyestuffs. There are some types of colours which will dye anything but a union. The other consideration should be the method by which the dyeing is made. On most types of rayon we find that a dyeing started at or near the boil and salted quickly, will give more colour on the rayon than when the same colour is dyed by the method of starting the dyeing at a lower temperature. On some types of machines it is not practicable to start dyeing at a boil, and on some others, it is. We know of plants securing excellent unions between low lustre rayons and mercerised cotton, by preparing the dye bath at the boil, and throwing in the dry material, running at the boil and salting after 15 minutes. In another case, this procedure is followed, with the exception that Glauber's salt is used, and the salt is added with the dye before the material is entered.

Types of Hose giving Trouble

Another type of hose which gives trouble more or less regularly is the ladies' plain mercerised cotton hose. Tiger stripes have been and still are periodical nightmares. We believe that in some cases when this occurs it can be slightly reduced and made to appear less pronounced by care in the dye-house, but rarely, if ever, entirely eliminated. If tiger stripes are caused by unevenly applied softening oils in the yarn, it may be improved by a good detergent and by boiling. Two tones in the same dye-bath occur more frequently on this type of hose than any other. This problem is allied with that of tiger stripes, in that in some cases there is little or nothing that can be done about it in the dye-house. However, this trouble is frequently caused in the dye-house and can be corrected there. Uneven heating in the dye machine is the first thing to be checked up on. Then again, the selection of proper type dyestuffs is important. Colours which level well and exhaust well without salt or with a minimum quantity of salt should be chosen. I believe the ideal way to dye this type of ladies' plain mercerised hose would be to use pocket type machines; to use carefully selected dyestuffs and quality grade scouring and penetrating oil; to start at low temperature, raise to the boil and boil vigorously during the entire dyeing period, and to avoid using salt.

One of the most common problems on those styles which cannot be boiled on account of sulphur dyed yarns or acetate yarns, has been spotted heels and toes occurring in very light shades. It has been my experience that this can be overcome by preparing the dye bath with the proper assistants, at approximately 170° F. and entering the dry material without previous wetting out. Of course, this method would not be practical in some type of dye machines. One of the fibres which has come into use more recently on fancy half hose is spun resisted silk. I have found that it is necessary to guard against any degree of alkalinity in the dye bath, in

order to work this yarn without staining. Even a small amount of alkali in the dye bath will frequently cause dyed resisted silk to change its tone, and will also cause natural or white to become badly stained.

Selection of Proper Colours

In thinking over fancy half-hose dyeing problems in a general way, I always arrive back at one point; and not only in half-hose, but in practically all other dyeing, we sooner or later find ourselves returning to this same point. That is, the selection of the proper colours for the work at hand. There are any number of dyes which have different properties of fastness and exhaust. There are union dyeing colours and those that dye one fibre and leave another clear. There are colours which will dye from the peroxide bleach bath, and colours which will work well in an alkaline degumming bath. There are also colours which will not do these things. To my mind, the selection of the proper colour is one of the principal solutions of all our problems, and closely allied to this is the selection of proper assistants to be used in dyeing. Then, of course the procedure or method is also important, but to my mind it is secondary to the selection of the proper type dyestuffs

British Enka Artificial Silk Co.

IMPROVED results were achieved in 1931 by the British Enka Artificial Silk Co., Ltd., the working profit of £34,546 comparing with a working loss of £72,139. But after providing for depreciation, fees and interest, and reserving £13,889 for unrealised loss on exchange, the net loss is £78,279, increasing the debit at profit and loss to £484,486. The net loss in 1930 was £166,607. The capacity of the works was slightly increased during the year, and a further moderate extension is approaching completion. The works were fully occupied, and the output was disposed of without difficulty at prevailing prices.

Solazol Colours and Related Products

Derivatives of Beta-Amino-Anthraquinone

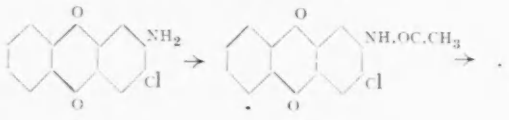
THE Solazol colours may be regarded as related to the anthraquinone and to the Naphthol AS colours. Whilst possessing the same degree of fastness as the Insoluble Azo colours, they are applied in a manner similar to the Indigosols and Soledon colours.

As an example of the synthesis of a Solazol dye, the well-known beta-amino-anthraquinone, chlorinated in the 3-position (I) is acetylated (II). The acetyl-amino-chloranthraquinone is then esterified (III), hydrolysed to the amino body (IV) and this is diazotised and coupled with Naphthol AS to give the Solazol colour (V).

The Solazols are applied to fibres bp dyeing, padding or printing from a neutral or slightly acid solution. On after-treatment in a hot acid oxidising bath the solubilising ester groups are split off and an insoluble pigment dye is formed in the fibre.

Methods of Preparation

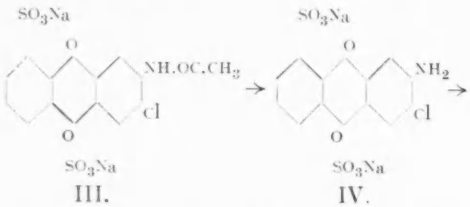
The first stage is the preparation of the sulphuric acid ester of beta-amino-anthraquinone or one of its derivatives. The amino group is protected by acetylation, and the resulting acetylamino-quinone is then treated with an alkyl sulphuric acid halide or chlor-sulphonic acid or oleum in a tertiary base, e.g., pyridine, quinoline, dimethyl-aniline, in the presence of a suitable metallic powder, e.g., zinc. On boiling with caustic soda the acetyl group is removed, and the ester may be isolated by salting out. An alternative method of preparing the esters of beta-amino-anthraquinone consists in boiling under reflux with ammonia the sulphuric ester obtained by reacting on anthraquinone-2-carboxylic acid with pyridine-sulphur-trioxide in the presence of a metal.



I. II.

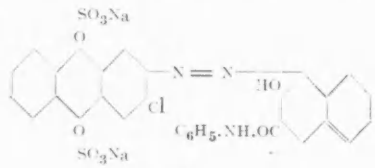
In the next stage the amino-group is diazotised without hydrolysis of the sulphuric acid ester group. This is accomplished by using only the theoretical quantity of sodium nitrate and hydrochloric acid necessary for diazotisation. The slightest excess of nitreous acid would hydrolyse the ester group and for the same reason the sodium nitrate must be added all at once so that there is no formation of the oxidising agent, nitreous acid, in the presence of mineral acid. Finally, the diazo-compound may be coupled with any passive component to give the Solazol dyestuff. If the passive component is a hydroxy-derivative, e.g., phenol, beta-naphthol, Naphthol AS or its derivatives, the latter is dissolved in caustic soda and a solution or suspension of the diazo compound run in slowly with constant stirring. After about an hour, the mixture is warmed and the dyestuff salted out. If the passive component contains an amino-group, e.g., beta-naphthylamine, the latter is dissolved in acid and the diazonium solution run in as before.

If to a solution of the sodium salt of beta-amino-anthrahydroquinone-9 : 10-disulphuric acid ester excess hydrochloric acid is added at 0° C. and the solution treated with the theoretical quantity (for diazotisation) of sodium nitrate, the



III. IV.

bright red colour of the diazo compound gradually disappears and the solution acquires a bluish colour. After several hours stirring at room temperature, the diazo compound has disappeared and the beta-amino-α : β-azo-anthrahydroquinone-tetra-sulphuric acid ester may be salted out. The amino-anthrahydroquinone-9 : 10 disulphuric acid ester may be used, not only as an active component, but also as a passive component. Thus, when used with diazotised para-nitranaline it yields a product dyeing reddish-purple but developing to a brown hydrolysis of the ester groups.



V.

In addition to the use of amino-anthraquinones in the above reactions, the amino-benzoquinones may be employed, and also the amino-anthranols. The latter are conveniently prepared by dissolving the corresponding amino-anthraquinone in oleum, and adding copper powder, the temperature being maintained at 20°-30° C. After stirring for several hours the product is poured into water and filtered. The amino-anthranol remains as a grey powder, insoluble in water, but dissolving in alkali with a brownish yellow colour. Finally, the sulphuric acid ester groups may be replaced by phosphoric ester groups, the acetyl-amino-quinone being treated with phosphorus oxychloride and copper powder in pyridine solution.

Extending the Uses af Nickel in Japan

A NICKEL information bureau is being established in Tokyo (Japan) by the International Nickel Co., Inc. Mr. James A. Rabitt, formerly Commercial Attaché to the American Embassy at Tokyo, will act as adviser to the Bureau.

Basic Intermediates for Dyestuffs—No. XLVII.

A Study of Some Acid Anilides.

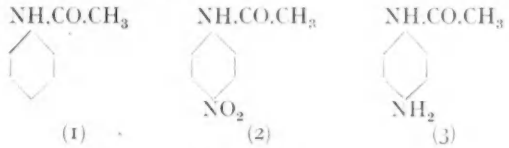
By "Consultant"

THE anilides are a class of compounds which well repay a little study from the commercial point of view since they are in great demand for the production of dyes of all classes, including both the compound azo dyes and the vat dyes. The most simple anilide is acetanilide (1), produced in quantities both for pharmaceutical and dyestuffs purposes. The digester for the preparation of acetanilide is made of cast aluminium, the interior surface of which has been "planished" in order to remove surface irregularities which readily accelerate corrosion; iron pans are definitely not of any use in acetylation processes. The reflux condenser is maintained so that the water can pass off; it carries with it a little acetic acid, but the majority is retained in the digester. The temperatures, measured in the centre of the reaction mixture are arranged as follows:—First ten hours 110° C., second ten hours 120-130°, next five hours up to 150°. After the latter period of five hours has elapsed the remaining water is distilled off together with an excess of acid that may have been present and the temperature is then gradually raised until it reaches 250° C. Fortunately, acetanilide is stable, and will withstand this temperature without appreciable decomposition. The quantities for a "run" in the usual type of plant amount to ten gallons each of glacial acetic acid and aniline, the latter being a good "white" oil, preferably WW. The acetic acid content of the glacial acid should not be below 98 per cent. When the reaction is over and the melt is at 250° C. it is run out through the bottom cock into water (100 gals.; maintained at 55° C.) and well stirred; the cooling and agitation cause the acetanilide to solidify in pellets which are easily drained and freed from excess moisture by centrifuging.

The Nitro-acetanilides

The bulk of dyestuff acetanilide is prepared for nitration, the objective being *p*-nitraniline. Thus, in nitration of acetanilide the conditions are focussed on the formation of the maximum yield of the para derivative, the ortho derivative being of comparatively minor value, and is often not recovered. Precise strengths of acids and a nitration temperature of less than 3° C. are essential conditions. The nitration may be done successfully in a silicon cast iron digester. The sulphuric acid (98 per cent.; ¼ ton) is run in, and is both stirred and cooled, either by external jacket or by brine coils. The acetanilide (1 cwt.) is sprinkled in with a scoop through the manhole, the temperature being kept as far below 30° C. as possible. The elevation of the temperature above 30° C. involves considerable loss my hydrolysis of the acetanilide and sulphonation. When all the anilide has gone into solution the manhole cover is replaced, the brine coils are turned full on until the temperature drops to 0° C. after which the nitration acid is run in. The composition of the acid is important, and may be obtained most easily by mixing equal parts of sulphuric acid of density 1.8415 and nitric acid at 1.4. This mixture contains 32 per cent. of HNO_3 and 49 per cent. of H_2SO_4; about 1½ cwt. of mixed acid are required, and it is run in slowly until the addition is complete, maintaining the temperature below 3° C. The stirring gear is kept in operation until the nitration is over, about five or six hours after the last addition of acid being required. When nitration is complete the reaction mixture is run out into a mixture of ice (¼ ton) and water (100 gal.) when the mixed anilides separate out and are filtered off and well washed with water. To separate the para and ortho isomers, they are stirred with five times their weight of water and made alkaline with caustic soda. The degree of alkalinity is such that litmus is just turned blue. On raising the alkaline mixture to the boil, the ortho-nitroacetanilide is rapidly hydrolysed to the corresponding nitraniline, but under the specified conditions the para compound is unchanged. Boiling is maintained for about five minutes, after which the mixture is cooled to 50° C. and the paranitroacetanilide well washed with water and dried. The yield is 130-135 lb.

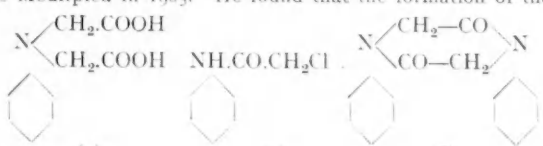
In the production of azo colours *p*-amino acetanilide (3) is valued for the stability it confers on the dyestuff normally it is prepared from the wet paste of the *p*-nitro acetanilide obtained in the foregoing process. The drying of the paste leads to a diminution in the yield of product. The reducing agent is iron and acetic acid. The paste of nitro compound is suspended in 40 gal. of water in an iron digester fitted with the usual gear and well stirred with iron borings (2 cwt.). When the mixture has been well stirred, 6 lb. of 80 per cent. acetic acid are added. The reaction soon starts in the boiling mixture and sufficient heat is disengaged to keep the mixture in a state of constant ebullition. The end-point is recognised by allowing a spot of the reaction mixture to fall on to a strip of filter paper; if unchanged nitro compound is present the "surround" of the spot will be yellow; when reduction is complete the area of solution surrounding the spot is colourless. At this point the solution is cooled to 70° C. and slowly made alkaline with soda.



(1) (2) (3)

Phenyl Glycine

Although phenyl glycine is not a true acid anilide, it is, nevertheless, obtained by the condensation of aniline with an acid. The text books say that phenyl glycine may be produced by the interaction of aniline and chloracetic acid. Actually this is not true in the commercial sense. When equal molecular proportion of aniline and chloracetic acid are heated together, the main product is ω-chloracetanilide and phenyliminodiacetic acid (4 and 5), while as a third product there is an appreciable percentage of the diketo-piperazine compound (6). The whole process was investigated by de Mouilpied in 1905. He found that the formation of these

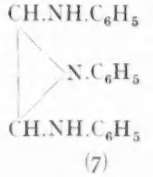


(4) (5) (6)

side products could be eliminated by the use of three molecular proportions of aniline. On the other hand the amount of phenyl glycine obtained did not reach the theoretical value on account of the formation of phenylglycine anilide; this compound may be hydrolysed to the phenyl glycine base, without much loss, and in one process is the means of isolating the phenylglycine from the reaction mixture. In one group of processes the reaction of aniline with formaldehyde and sodium cyanide is made use of:—

$$C_6H_5.NH_2 + H.CHO + NaCN = C_6H_5.NH.CH_2.COONa + NH_3$$

either as the direct process shown by the equation above or by first combining the formaldehyde with two molecules of aniline to obtain methylenedianiline, and then treating this with sodium cyanide to give the phenylglycine. Another group of processes depends on the interaction of aniline with trichlorethylene and the hydrolysis of the product so obtained. The key substance in this synthesis is often referred to as Sabanajeff's base, since it was discovered by this investigator.



(7)

To prepare it, trichlorethylene is heated with three molecular proportions of aniline, when the base (7) is formed. This is hydrolysed to phenylglycine anilide and thence to phenylglycine itself. Actually, of course, the intermediate compounds are not isolated; in the original process of Levinstein and Imbert the trichlorethylene was heated together with rather more than twice its weight of aniline in three times its weight of water at 180° C. in an autoclave for 24 hours, after which the mass was filtered.

The Examination of Dyed Furs

A New Filter Paper Test

DR. A. GOLDBERGER (*Cuir Tech.*, 1932, p. 98) considers that an examination of dyed furs, in cases of suspected dermatitis, in ultra-violet light forms a useful supplement to the chemical tests as already published by Cox, Callan and Stafford. A number of the Ursol fur dyes have been examined both in the solid state and in alcoholic and aqueous solutions. These colours are divided into two groups:—(a) Those fluorescing in the solid state such as Ursols D, DB, DBB, RH, NZ; and (b) those fluorescing in solution only, such as Ursols P, P base, AL, SA, Olive 3G.

The table given shows the fluorescence obtained when examined in both neutral, acid and alkaline alcoholic solutions, and also when the solution is impregnated on filter paper. A strip of filter paper 1 cm. wide is dipped in the solution until about 2 cms. have become wetted. The paper is then carefully dried over a warm asbestos hot plate, and examined. The best visibility of any fluorescence is when the paper is just dry. Usually the whole of the surface of the paper which has been wetted becomes fluorescent, although in certain cases the phenomena is complicated. For instance, an aqueous solution of Ursol SA (1 :10000) gives two distinct layers, the soaked part showing a yellow fluorescence, and that moistened by capillarity a bright violet. In another test, the paper from a solution of 0.01 gr. Ursol D and 1 gr. Ursol 4G per litre gave three layers when examined. The soaked portion showed "black" owing to its heavy charge of Ursol 4G which is non-fluorescent, then a brighter but still opaque band, and finally a limited band of a very bright violet fluorescence. The test indicates that the presence of non-fluorescing Ursols does not mask the presence of others, while it also indicates the differences in degree of dispersion of these substances in water.

In the examination of suspected furs, the material cannot be examined directly owing to the fluorescence given by some undyed furs themselves. Thus, white rabbit hair gives a strong violet fluorescence. Further, natural and added greases will interfere in a similar way. The method proposed is to extract the separated fur with ether, and then extract the fat-free material with weak acid in the usual way. This acid extract is made alkaline with sodium bicarbonate, and the filter paper soaked in this solution. A negative reaction is, of course, no proof that the fur is undyed, as some of the Ursol colours are non-fluorescing, but it so happens that the most dangerous substance liable to induce dermatitis, i.e. *p*-phenylenediamine, is the base of Ursol D, and this latter is one of the most fluorescent. Incidentally, the strongest fluorescence is usually given by the alcoholic solution of the dyestuff.

Product.	Alcoholic Solution.			Aqueous Solution.			Sensitivity of Test on Filter Paper.
	Neutral.	Alkaline.	Acid.	Neutral.	Alkaline.	Acid.	
Ursol D	Violet	Intense violet	Pale grey-blue	Very pale violet	Violet	Nil	1 : million at *pH* 6.7—12.0. 1 : 10,000 at *pH* 1.7.
Ursol DB and DBB ..	Do.	Do.	Do.	Do.	Do.	Do.	1 : million at *pH* >7.
Ursol RH and NZ ..	Do.	Do.	Do.	Do.	Do.	Do.	1 : 100,000 at *pH* >7.
Ursol P	Pale violet	Intense violet	Nil	Nil	Nil	Nil	1 : 100,000 at *pH* >7.
Ursol P base	Intense violet	Intense violet	Nil	Pale violet	Pale violet	Nil	1 : 100,000 at *pH* >7.
Ursol AL	Very pale violet	Violet	Pale grey	Nil	Nil	Nil	1 : 100,000 at *pH* >7.
Ursol SA	Pale brick red	Yellow	Pale brick red	Pale grey	Yellow	Nil	1 : 10,000 at *pH* >8.
Ursol Olive 3G	Violet	Blue	Nil	Nil	Nil	Nil	1 : 10,000 at *pH* >6.4.

Bulmer Rayon Co. Ltd.

SEPARATE meetings of the shareholders of the Bulmer Rayon Co., Ltd., were held in London on March 30. Mr. A. J. Pryor, who presided, said the reason for his occupying that position was on account of Sir William Bulmer having retired from the company through force of circumstances. Certain interests in the company, he proceeded, had deemed a liquidation the only satisfactory conclusion to all that had taken place, but they were compelled to recognise that the voting power of the company rested with the Deferred Ordinary shareholders, who in no circumstances wished for liquidation. The Preference shareholders were convinced that only 9d. per share would be realised, and this was problematical should liquidation ensue, inasmuch as the company was a defendant to an action at law which was regarded by prudent people as uncertain in its outcome. An offer had been made to the board of a controlling interest in Stedman, Crowther and Co., Ltd., metal merchants. Its capital was £20,000, £15,000 of which had been issued.

The dividend which would be declared by Stedman, Crowther and Co., would be available for the Preference shareholders. In the circumstances, it would be reasonable now to assume that substantially more than 9d. or 1s. would be secured for the Preference shareholders, while at the same time there would be a certain value for the Deferred Ordinary shareholders. Resolutions authorising the directors to enter into an agreement for the sale of the assets of the company were carried by the requisite majority.

Czechoslovak Case in Industry

IMPORTED casein is reported to have considerably affected the Czechoslovak casein industry through a decline of prices to a level below domestic production costs. An attempt is being made by Czechoslovak producers to secure limitation of imports.

Recent Dyestuff Patents

The following information is prepared from published Patent Specifications which may be obtained from the Patent Office, 25 Southampton Buildings, London, W.C.2, at 1s. each.

363,668-9. DYES. D. A. W. Fairweather, J. Thomas, and Scottish Dyes, Ltd., Grangemouth.

363,668. The disulphuric esters of anthraquinone-anthrahydroquinone-dihydro-azine and its substitution products are treated with a reducing agent, or tri-sulphuric esters of anthrahydroquinone-anthanolazine and its substitution products are treated with acids to obtain the sulphuric esters of anthraquinone-anthranol-dihydroazine.

363,669. The magnesium, lithium, ammonium, methylpyridinium and calcium salts of tetrahydro-dianthraquinoneazine tetrasulphuric ester are obtained by treating the potassium or sodium salts with the corresponding chlorides, acetates, or perchlorates of the desired basic radicles.

364,087. DYES. J. Y. Johnson, London. From *I. G. Farbenindustrie*.

A nitro-dibenzanthrone is treated with chlorine or an agent supplying it in the absence of a diluent or in the presence of an organic diluent other than phthalic anhydride or a halogenated derivative. The halogenation is continued until the nitrogen is reduced below 0.5 per cent., or alternatively the part having a nitrogen content less than 0.5 per cent. is separated from the remainder. Suitable chlorinating agents include sulphuryl chloride, and benzoyl chloride.

364,141. DYES AND INTERMEDIATES. Imperial Chemical Industries, Ltd., London, W. W. Tatum and R. F. Thomson, Earl's Road, Grangemouth.

Anthraquinone is halogenated in α-position or its halogen derivatives are halogenated further in the presence of sunlight or other actinic light, with or without halogen carriers and solvents, and converted into amino derivatives.

Dyestuffs Supplement

May 14, 1932.

Current Topics

The Textile Institute

In his presidential address at the annual meeting of the Textile Institute at Manchester on April 20, Mr. G. Garnett said he was increasingly convinced of the necessity for the information bureau and the collection of yarns and fabrics which the Institute proposed to set up. In his opinion, it may take years to establish the scheme effectively, but the thoroughness with which the responsible committee is applying itself to the task gives promise of considerable success. With the support of the trade organisations, many of which were already sympathetic to the movement, the Institute would be able to show that this information would be of great advantage to its own members and would merit the support of Government Departments, municipalities and other public bodies. Mr. Garnett also spoke of the benefits derived by the cotton industry from its participation in the British Industries Fair, but he expressed some doubt as to whether the woollen trade lent itself to such an open display of design and colouring. The Institute Council has decided to proceed with a scheme for national certificates and diplomas in textiles, and will place a proposition to that effect before the Board of Education. Mr. Garnett has been re-elected president of the Institute for the ensuing year.

Fastness of Dyes to Vulcanisation

Some eighty dyes belonging to different groups of textile material coated with rubber have recently been subjected to tests to determine their fastness during vulcanisation. This investigation is reported upon by A. V. Sourovaia in " Rev. Ind. Text. URSS " (1931), No. 6. The test pieces were subjected to hot vulcanisation at 132°-134° and 2.5 atm. pressure for three hours and to cold vulcanisation with sulphur chloride. The vulcanised pieces were then classified according to five degrees of fastness ranging from " complete change of shade " (1) to " no change " (5). The conclusions drawn are that hot vulcanisation lightens the shade in all groups of dyes except the acid dyes and those which are formed on the fibre, whilst cold vulcanisation results in a less regular change of shade for the different groups of dyes. The vulcanisation mixtures do not themselves inflence the change of shade, nor does the presence in the vulcanisation furnace of benzene, petrol and oil of turpentine vapours. Sulphur chloride leads to tendering of the treated fabric. Neither the layer of rubber with which the fabric is coated nor vulcanisation increases the fastness of the colour to light. The fastness of the substantive dyes, which have an average fastness of 3 to cold vulcanisation, varies from 1 to 5 for hot vulcanisation; the fastness of the basic colours varies from to 3 to 5 for both types of vulcanisation; the indanthrenes give a fastness of 3 to hot vulcanisation and of 3 to 5 to cold vulcanisation. The fastness of the sulphur colours to hot vulcanisation is 3, except that of Sulphur Black TF which is 5, and to cold vulcanisation is 3 to 5, except that of Sulphur Green which is 3; whilst the fastness of the acid colours and of colours formed on the fibre ranges from 4 to 5.

A New I. C. I. Acid Dyestuff

Lissamine Violet 5BS, a new addition to the I.C.I. range of acid dyestuffs, is particularly suitable, in combination with other acid dyestuffs, for the production of navy blue and other mode shades where fastness to light is not of importance. It possesses very good level-dyeing properties and does not stain cotton or artificial silk effect threads, which makes it of special value for the dyeing of piece goods. It is of moderate fastness to light, acids and alkalies, but possesses more than average fastness to washing, and on account of its excellent penetrative properties, is eminently suitable for dyeing navy blue shades on wool and fur felt hat bodies. Lissamine Violet 5BS is practically unaffected by the presence of iron or copper in the dyebath and is therefore suitable for use in metal dye vessels. The colour is particularly suitable for the direct printing of wool, silk and tin-weighted silk and may be used as a ground shade in pale and medium shades for subsequent discharge to a white with either Redusol Z or Formosul CW. It possesses excellent affinity for the wool fibre when dyed from a sulphuric acid dyebath, also very good level-dyeing properties, and if necessary, the dyebath may be fully exhausted by an addition of sulphuric acid towards the end of the dyeing operation.

Two New Geigy Colours

The Geigy Colour Co., Ltd., has recently introduced a new direct colour, which is fast to acid and alkali and possesses an exceedingly bright, pure and bloomy orange shade, second to none amongst direct dyes. Diphenyl Fast Orange 3RS is suitable for dyeing cotton and artificial silk of every description, with the exception of acetate silk, which is left a pure white. This colour can be adopted also for unweighted silk wherever good fastness to acids and alkali are required. Dyed at the boil upon union material level shades are obtained, whilst at medium temperature the wool is stained much lighter; with an addition of Erional L the wool remains almost white. On half silk (gloria), applied in the normal way with Glauber salt, Diphenyl Fast Orange 3RS dyes silk weaker than cotton, whilst when dyed with soap and soda the silk is left a pure white.

Diphenyl Fast Pink G gives a pure, bright pink shade and is suitable for dyeing cotton of every description and chiefly artificial silks (acetate silk excepted) also for mixed fabrics consisting of cotton and artificial silk, on which material solid shades are obtained. This colour is unaffected by artificial silk of varying quality and it is also exceedingly well dischargeable with hydrosulphite.

International Dyestuffs Agreement

THE directors of Imperial Chemical Industries, Ltd., are convinced believers to the long range wisdom of international agreements as instruments of world rationalisation in industry. In his address to the shareholders on April 14, Sir Harry McGowan said that in pursuance of this policy, the directors had recently concluded an agreement, following protracted negotiations, with the continental manufacturers of dyestuffs. For some years arrangements have existed between French, German and Swiss makers under which they co-operated with one another and marketed their products in an orderly manner. For some time the British dye industry was regarded as not having proved its permanence. Since 1927, when the industry was brought within the ambit of the I.C.I. merger, no effort has been spared to increase its competitive power, to extend the variety of its products, to introduce new and improved dyestuffs, and to develop a service to the dyestuffs user which should meet his every requirement. In these matters the directors claim to have been remarkably successful. Financial results have not been allowed to interfere with our efforts, and he confessed that, during this process of improvement, the monetary results of their operations had not been economic. But the position has slowly grown better, and to-day they are satisfied with the virility of this industry. Their international position has been greatly strengthened by the agreement in question. It will not diminish their normal incentive to improve existing products or to widen their range by new inventions. Research and development will proceed as hitherto. It will still permit of colour-users obtaining from continental makers dyes not produced in this country.

The Glazed Kid Industry

THE present financial position has given an extraordinary fillip to the glazed kid industry in this country. This branch of the leather trade has been making steady progress during the last nine years and the returns show that the British manufacturers have been securing an increased share of the trade of the country. In the slump of 1920 which caused the closing down of several glazed kid tanneries, the rest of the manufacturers were obliged to turn their attention to the production of coloured kid, in order to meet the requirements of the customer. Slow but sure progress was made in this direction, and the present opportune moment has resulted in a great increase in the output of this kind of leather in Great Britain. Several tanneries which have been closed down since about 1920 have re-opened during this year, and it is expected that production on a large scale will soon be attained. The older established glazed kid tanneries are increasing their output to meet the new demands, and workpeople who left the industry when trade slumped so badly some years ago are being re-instated. There is actually a shortage of skilled people. One of the leading producers has increased his daily input of raw goatskins from 2,800 per day to 5,000 and every machine in this factory is working at full capacity. Some manufacturers are handicapped in the finishing of coloured leathers but assistants are being trained to cope with the situation. Larger quantities of bichromate of soda, hydrochloric acid, hypo and red arsenic will be consumed as a result of these increased activities.

Dyestuffs Licences for April

THE following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during April has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee: The total number of applications received during the month was 329, of which 254 were from merchants or importers. To these should be added one case outstanding on March 31, making a total for the month of 330. These were dealt with as follows:—Granted—309 (of which 303 were dealt with within seven days of receipt); referred to British makers of similar products—13 (of which 12 were dealt with within seven days of receipt); outstanding on April 30—8. Of the total of 330 applications received, 315 or 95 per cent. were dealt with with seven days of receipt.

American Textile Colour Cards

An Increase in Foreign Membership

ACCORDING to the report presented at the annual business meeting of the Textile Colour Card Association held in New York, the Association's new members gained during the past year included a large number of foreign firms, indicating the increasing interest abroad in the Association and the American cards. These new foreign members are located in Australia, Austria, Belgium, Canada, Cuba, England, France, Germany, Italy, Phillippine Islands, Scotland and Sweden. During the year the Association manufactured 51,607 cards, including silk, wool, shoe and leather seasonal, hosiery standard and seasonal, supplementary mid-season cards and special cards showing the basic colours for retail buying, as promoted by the National Retail Dry Goods Association.

An active part was taken by the Association in helping to form the Inter-Society Colour Council, whose aims and purposes are to stimulate and co-ordinate the work being done by the various societies and associations leading to the standardisation, description and specification of colour, and to promote the practical application of these results to the colour problems arising in science, art and industry.

Rich Bordeaux and mellowed Burgundy tones will occupy an important place in the collection of fifty new colours portrayed in the 1932 Autumn Woollen Card, soon to be issued by the American Textile Colour Card Association. The mahogany range, including rhum and spicy hues, is also cited as another feature in autumn colour fashions. Despite the growing style significance of reds, the brown family shows every indication of occupying an even stronger position than it did last winter. In addition to the reddish mahogany gamme mentioned above, a dark tête de nègre range is represented, the darkest tone being described as an ebony brown, almost black.

A deep beetroot shade, more purplish in cast than the vintage reds, is included, and rich, clear tones of red, including a smart raspberry hue, also figure prominently. New interpretations of tile shades, some inclining to red and terra cotta, others bordering on brown, are included in the group. In the beige range, preference is expressed for sandy and grège tones, as well as a yellowish beige of the honey type. Blues for early autumn embrace a wide range.

Increased Uniformity In Leather Dyeing

By D. WOODROFFE, M.Sc., A.I.C.

LEATHER is an extremely variable material. No two skins are alike in appearance, texture, substance and colour. When packs of skins are dyed, there are always variations in shade and depth of shade as a result. In addition, there are differences in texture, colour and substance in the individual skins, which render it extremely difficult to ensure the production of level dyeings without special precautions, such as the careful selection of the dyestuffs and the adoption of different expedients designed to assist in this particular. In spite of the various devices used for obtaining level dyeing, leather dyers have not been any too successful in securing the results which they aimed at. Leathers have deteriorated in quality owing to reasons beyond the control of either the leather dyer or the tanner. Farmers and stock-breeders have adopted certain methods or stock-rearing and breeding which are detrimental to the texture of the skin, and since the pelt is only a by-product, it is unreasonable to expect them to pay much heed to the complaints of the leather manufacturer.

The chief methods employed in leather dyeing are the tray, paddle and drum methods. The tray method is a hand-method dating back for a considerable period and still used on certain high-priced speciality leathers; the drum and paddle methods are employed more commonly. No essential changes have been made in the shape or general form of drums or paddles, but there have been several minor improvements of value to the dyer.

A New Form of Paddle

A new form of paddle was recently designed by M. C. Lamb for carrying out the dyeing operation in a more scientific manner. It is completely enclosed, thus ensuring a lessened liability of cooling during the process. The cover is fixed to the paddle and it may easily be opened to permit of the inspection of the skins or leather during dyeing. The chief feature of this new paddle is the provision of steam and cold water supplies below a false bottom. The latter is moveable, but it is not suggested that it should be removed for purpose of dyeing. The arrangement enables the paddle to be filled with water at any required temperature, but its chief value lies in the possibility of heating up the dye liquor during the actual dyeing of the leather. This is a common practice in textile dyeing where the wool, cotton or other material is placed in cold solutions of dyestuffs, particularly basic dyes. After some time, the temperature of the liquors is gradually raised to 160° F. by steaming up. Textiles are capable of standing the presence of live steam passing through the liquor without any danger of being injured, but if the same procedure was adopted in the case of leather dyeing, the leather would be spoilt. Leather dyers have always exercised the greatest possible care in warming dye liquors in which there was any leather being dyed.

Lamb's new paddle arrangement permits of the warming up of the dye liquor even whilst the leather is in process of being dyed. This is a great improvement on the older method. The dye solution is at its strongest at the commencement and therefore it is most likely to be fixed readily being dyed. This is a great improvement on the older to fix the dyestuff, in fact it will absorb the dye with great avidity. Experience has shown that less even colouring effects are obtained if the initial rate of fixation of the dyestuff is high. By commencing the dyeing in cold or tepid solutions, the fixation of the dyes is restrained at the commencement of the dyeing process when the leather is in a condition to absorb the dyestuff with greatest avidity. The temperature recommended for starting the dyeing is 95° F., and as the dyeing proceeds the temperature is gradually raised to 130° F. by allowing steam to pass into the paddle through a pipe provided for the purpose below the false bottom. It is claimed that the use of this paddle produces greater uniformity in shade of the dyed leather and that there is an almost complete absorption of the dyestuff. The paddles are fitted with a perforated copper tray into which the dissolved dyestuff can be poured. By this device, contact of the strong dyestuff solution with any of the skins is avoided.

There have also been some developments in the construction of dyeing drums. The old type of drum with the ordinary cog drive was extremely noisy and was a constant jar on the nerves of those in charge. The adoption of enclosed geared drives mounted on ball bearings has eliminated most of the noise in the latest pattern drums. The shape has also been altered to octagonal and the drums are provided with specially arranged shelves which tend to throw the liquor in the drum from one side to the other and so mix it more efficiently.

These improvements in leather dyeing machinery should be welcomed by dyers, since there has been so little change or improvement since drums and paddles were first introduced for leather dyeing. With greater efficiency, it is possible to exhaust the dye liquors almost to completion with a consequent economy in dyestuff. Much more important than any economy is the increased possibility of reproducing results, for it is an alarming fact that one can never be certain of repeating a particular shade which may have sold well. Even the textile dyers will not guarantee that a repeat dyeing will be an exact match, in shade and depth, of any previous dyeing. With all our advances in science and in practice this is surely a serious deficiency. The causes are not far to seek. The leather dyer will inevitably blame the variations which occur in the tanned leather and which in many cases are quite beyond his control. This is undoubtedly a frequent source of trouble, but it is not the only one. Many of the variations are within the province of the dyer. He can select the size of his pack where there are large bulks to be dressed, the large skins being sorted out and dyed separately if this is thought desirable.

Leather dyers usually employ drums or paddles. In some works there is only one size of drum and perhaps two or three sizes of paddles. The dyer responsible for the process informs the workpeople how much water to use in vague terms, such as one-third, one-quarter or half-full in the case of drums, but no variation can be made in the case of paddles. Small packs of skins can be dyed in small paddles, but the proportion of water in these bears no comparison with the weight or number of skins to be dyed in them. From 25 to 35 dozens of small skins would be dyed in a large paddle of 300 gal. capacity, 20 dozens in a paddle of 230 gal. capacity and three dozens in a small paddle of 80 gal. capacity. It is evident from this example, which is taken from an actual works, that the results obtained in dyeing an experimental lot of leather of three dozens in 80 gal. of dye liquor cannot be expected to bear any simple relationship to those which will follow in a 300 gal. paddle for, say, 30 dozens skins. The amount of water employed in dyeing the experimental batch is two and a half times as much as that used on the bulk lot. Can it be wondered at when the results obtained on the sample lots are not reproduced on a large scale?

Amount of Water Used in Processing

Leather dyers rarely, if ever, pay attention to the actual amount of water used in processing the skins. Of course, one cannot alter the amount of water in a paddle, but it can be accurately adjusted in a drum, and they omit to do it. On the Continent, the amount of water is invariably calculated exactly in proportion to the weight of leather to be dyed or processed. There is no reason why laboratory tests on the small scale should not be reproducible on the larger scale if every variable factor is similarly adjusted. The percentage amount of water should be the same in each case, and in most leather dyeing in Great Britain this factor is only roughly adjusted. The writer has used dyeing recipes which had been carefully worked out for bulk lots of 1,000 lb. of leather, and applied them successfully to the dyeing of one skin weighing less than 1 lb. The leather manufacturer must pay more attention to the question of the concentration of his various liquors and solutions if he is to ensure a greater degree of uniformity.

Another variable feature in leather dyeing is the actual temperature of the dye liquors. The usual practice consists

in running up the dye paddle or drum to the required depth with water at 128° F. for vegetable tanned leathers or at 130-140° F. for chrome tanned leathers. The wet skins are then entered from the mordanting or other preliminary process and as they are usually cold or at best merely tepid, the dye liquor is cooled down a little. The result is that, except by the merest coincidence, different batches of leather are never dyed exactly at the same temperature and consequently the results obtained with the same dyestuffs are different also. This variation in the actual temperature of the dyeing accounts for the patchy dyeing and uneven results obtained in some dyeings and not in others. Lamb's suggested arrangement for warming up the liquors in a paddle would undoubtedly obviate this variation, but there would still be the same variations in drum dyeing. Some dyers make an allowance for the cooling effect of the cold skins, but it is generally an intelligent guess and is not scientifically accurate. Leather manufacturers and their staffs should aim at more scientific accuracy, for it is not a difficult or impractible proposition to consider the exact adjustment of the temperature after the skins have been entered into the paddle or drum.

The writer has shown that the shade produced by acid dyestuffs on leather depends on the actual *pH* of the leather. So far as he is aware, no-one is controlling or adjusting the *pH* of the leathers or the dye liquors. The *pH* of leather has been shown to vary within a range of 2-5, which is in itself quite sufficient to produce appreciable variations in depth of colour with many acid dyestuffs. The *pH* of a vegetable tanned leather depends on the nature of the tanning material, the mode of tannage, the acidity of the tan liquors and the addition of acids, bleaching materials, synthetic tannins, fats, soaps, etc. The preliminary processing of the leather, *viz.*, stripping with alkalis, retannage and souring, alter its *pH* value. Here again the leather dyer frequently varies his process by his failure to use the same percentages of water, tanning material, acid and other chemicals necessary for the preparatory treatment. Thus some batches of the leather enter the dye liquors at a *pH* of 3.0 whilst others may be at 3.5, 4.0 or 4.5. There is an entire absence of any control in this particular. Moreover, when the dyer is ready for adding the necessary amount of acid to the dye liquor, the quantity is not determined by the actual requirements of the leather but generally by the size of the paddle or drum and the amount of dyestuff which has been employed. It would be a simple matter to control the *pH* value of the dye liquor with the Wulff colour strips, whereby the figures can be obtained accurate to 0.2, and then acid could be added in sufficient amount to reduce the value to any particular figure thought desirable, instead of the present haphazard methods.

The Lustre of Acetate Rayon

By E. POYNER

The following article is reprinted, by permission, from the April issue of "The Silk Journal and Rayon World." It deals with a particularly interesting subject in view of the wide application which rayon now enjoys as a textile material.

ALTHOUGH the demands of fashion have resulted in much attention being given to the subject of the lustre of the various kinds of rayon, precise information is still unfortunately the exception rather than the rule. One of the original differences between silk and the rayons lay in the lustre, the metallic sheen of rayon standing in sharp contrast to the subdued lustre of silk. When the great bulk of the output of rayon was used in the form of knitted fabric for underwear, the distasteful appearance due to the lustre could, to some extent, be discounted. The large difference in cost between rayon and silk hosiery was, until the great fall in the price of natural silk, probably the saviour of rayon in this particular application. The lower cost of silk, however, forced rayon manufacturers to consider ways and means to produce materials more nearly approximating to silk in lustre. This activity is well reflected in the patent literature, which contains many references to methods for obtaining the subdued lustre desired. It is probable that the development and extensive production of woven fabrics composed entirely of rayon for outer garments drove home the need for the reductions in lustre. Highly lustrous fabrics have a strictly limited application, even if made from the older textile materials. As almost unconsciously the fine fabrics of the past are selected as the standards against which new productions are compared, it is natural that the metallic lustre of rayon should have been considered a serious defect.

Factors Governing Lustre

In this article it is proposed to deal exclusively with the lustre of acetate rayon. Acetate differs fundamentally from the other varieties of rayon and responds differently to treatment designed to cause it to imitate silk in appearance. Its lustre as ordinarily produced is much less pronounced than that of viscose, and, on the whole, it may be claimed that it is subjected to closer control in manufacture and subsequent treatment. The varieties of rayon which consist of regenerated cellulose necessarily behave somewhat similarly to cotton and other forms of naturally occurring cellulose. They are not so readily attacked by chemical agents as an ester like cellulose acetate. Their physical state of aggregation is less easily altered than that of the esters which are probably of greater molecular complexity. Lustre appears to be governed mainly by microscopic structure rather than by the atomic or molecular architecture, since all the evidence points to optical explanations. As the ultimate structure of the material is revealed only by radiations of wave lengths far shorter than those constituting visible light, it would not appear to be directly concerned in the question of lustre.

The susceptibility of cellulose acetate to attack by chemical agents affords the simplest means of altering its lustre. When acetate rayon was first placed on the market it was necessary to educate the users of it as to the correct methods of laundering in order to keep the lustre unimpaired. Boiling soap solution readily reduces the lustre of acetate rayon. Weak solutions of caustic alkalies act with still greater rapidity. It is not necessary even to boil in soap solution if the temperature is maintained at just below the boil for an extended period. At first sight it would appear that this action is purely chemical, but investigation shows that although chemical action does occur the reduction in the lustre is mainly due to a physical process. That some chemical action is involved is definitely proved by the fact that the outer portions of the filaments of acetate are converted into hydrated cellulose. Direct cotton dyestuffs which have no action on acetate will dye cellulose acetate which has been boiled in soap solution. Cross sections of such material show clearly under a magnification of 300 to 400 diameters an outer skin of dyed cellulose hydrate, the body of the filament remaining quite colourless. Cellulose such as viscose or cupprammonium rayon on dyeing in the same bath is completely penetrated by the dyestuff, the cross section showing uniform dyeing. If the boiling with soap is continued for a sufficiently long time it is possible to convert the acetate wholly into hydrated cellulose, which has approximately the same lustre as the original acetate.

An Optical Explanation

Further examination of the cross sections of cellulose-acetate filaments which have been delustred by means of boiling soap solutions shows discontinuities in the structure which are not seen in the cross sections of lustrous material. Examination by means of the microscope of the surface of delustred yarn also shows these discontinuities. Lustrous material reflects incident light in a regular manner, usually known as specular reflection, whilst material showing discontinuities of dimensions not widely different from the wave lengths of the incident radiations scatters the light in all directions and consequently appears white. Ordinary glass provides an excellent illustration, reflecting light regularly as an ordinary polished surface. If the surface be ground with a fine abrasive, it assumes a white appearance

and a matt surface obtained in this way owes its properties to optical scattering. In the working of optical glass all the stages from practically 100 per cent. specular reflection to complete scattering are observed. The best explanation of the reduction in lustre due to boiling in solutions of soap or alkalies is that the attack of the chemical substances permits of the introduction of water which destroys the continuity of the colloid. In order to reduce the lustre of cellulose acetate only a very slight chemical attack is necessary, since the effect is essentially a surface phenomenon. The strength of fully delustred acetate is only slightly less than that of lustrous material.

This optical explanation of the reduction of the lustre of cellulose acetate receives support from the consideration of the method of restoring the lustre. Material of which the lustre has been impaired by washing in boiling soap solution may be relustred by means of acetic acid or certain other swelling agents. The fabric is soaked for a time in acetic solution of 15 per cent. to 25 per cent. concentration, and is allowed to dry at normal air temperature without washing off. When dry its temperature is raised for a short time to 80° C. to 100° C. On scouring the material is then found to have recovered its lustre. The swelling which takes place restores the continuity of the substance, excluding at the same time the water which was the cause of the dispersion of the colloid. For the success of this operation it is necessary to use a swelling agent which evaporates from the fabric less readily than water. Acetone solutions, although acetone is a more powerful swelling agent and solvent for cellulose acetate than acetic acid, do not relustre acetate when applied in this way, because the high vapour pressure of the solvent causes it to evaporate before the water. It is possible that the mercerisation of cotton may be partly explained on somewhat similar lines. The action of the alkali causes the conversion of the natural cellulose into the hydrated form with considerable swelling.

Treatment with Soap or Alkalies

The ease with which the lustre of cellulose acetate may be altered by means of solutions of soap or alkalis is not as advantageous as at first sight it might appear. In the first place it demands that acetate must be more carefully laundered than other textile materials. A gradual reduction in lustre would not be a very serious matter if it were not accompanied by other and graver troubles. The difficulty lies in the instability of the low lustre produced by soap solutions too hot for proper laundering. It is found that in ironing such delustred fabric all the parts which are wetter than their neighbours become relustred, and the cloth assumes a patchy appearance. Even if it is again boiled in soap solution in an endeavour to make the fabric of uniform low lustre, the parts which relustred on ironing still show faintly. This action of the hot iron in removing water of dispersion is one of considerable interest. It is possible to iron delustred fabric without reviving the lustre, but the precautions necessary are such that they would never be adopted in the practice of ordinary domestic laundering. These considerations proved ultimately that the reduction of lustre in this simple way was not a commercial and practical proposition, since many complaints would be likely to arise.

Patents have been granted for the addition to baths of various substances claimed to prevent the loss of lustre of cellulose acetate on boiling, but all are of doubtful efficacy and of little or no application outside the factory. Acetate may be delustred in numerous other ways involving chemical or mechanical treatment, but no method sufficiently good to warrant extensive adoption has been found. The more recent patents indicate that attention has been focussed upon methods essentially physical in nature. Incorporation in the dope of white mineral pigments gives yarns of lower lustre than those produced under ordinary conditions. The pigments must be prepared with great care or trouble with the spinning jets is experienced. Pigmented material is considerably weaker than ordinary lustrous acetate, the fall in strength being sometimes as much as 40 per cent. The low lustre of such yarn is not subject to change on laundering like that of material delustred by means of boiling soap solutions. With this process the main difficulty seems to be the low efficiency of the pigments as reducers of lustre. To produce as low a lustre as is desired generally requires more pigment than can conveniently be incorporated in the dope and the strength of the yarn falls as the amount of pigment is increased. It has been urged that in the use of yarns in which low lustre is due to pigmentation there are difficulties in processes such as warping and weaving.

An Ingenious Method of Reducing Lustre

An ingenious method of reducing the lustre of acetate yarn appears in the recent patent literature. The pigments, which may be oxides or insoluble salts, or even china clay, are prepared in the form of fine dispersions or colloidal solutions from which the acetate takes them up. The process may be accelerated by the use of swelling agents. The electric charges on the particles in the suspensions undoubtedly play an important part in this process. The idea has been extended to the mordant dyeing of cellulose acetate.

The intrinsic differences between silk and the various kinds of rayon are so extensive, and so well marked that the wisdom of trying to make rayon more nearly resemble silk may be seriously questioned. However its appearance may be changed rayon is never likely to be mistaken for silk. Further, rayon now enjoys an exceedingly wide application and is thus able to take its place, with its distinctive properties, among the regularly used textile materials. There is little to be said in favour of processes designed to make acetate rayon like silk in lustre, when the result of the treatment is to produce an inferior article, probably at an enhanced price.

Scottish Dyestuffs Market

Increase in Costs

ON the whole business in Scotland has shown a very slight improvement during the past month in the dyeing, finishing, bleaching, and similar lines, and although it is still quiet the level of employment has risen slightly. In the south, the tweed position is still difficult, orders for next winter only coming in slowly and merchants being shy of adding substantially to their stocks. As a result factories remain on short time. At a recent Chamber of Commerce meeting at Hawick, many questions relative to the border wool trade were discussed, reference being made to the difficulty in some cases of obtaining payment of accounts due by customers abroad, owing to the instructions imposed by the governments of the countries concerned.

A great increase in all costs caused by present methods of sampling and selling was referred to by the president. Although it might be impossible for various reasons to get all the firms joined together it was thought that a number of them might do so with advantage and so reduce expenses. This dealt with the matter as far as sales were concerned, but on the sampling side the question was more difficult.

Amalgamated Society of Dyers

Annual Conference

THE annual conference of the Amalgamated Society of Dyers, Bleachers, Finishers and Kindred Trades was opened at Blackpool on May 7, under the presidency of Mr. J. Mularchy, who, in his presidential address, said the past year had not been a bright one for the dyers and bleachers. A civic welcome was extended to over 150 delegates.

A resolution was passed urging the Government to abolish the means test in connection with transitional benefits, and officials were instructed to abstain from granting permission to employers to exceed the maximum working week of 60 hours until the whole of the unemployed members were absorbed.

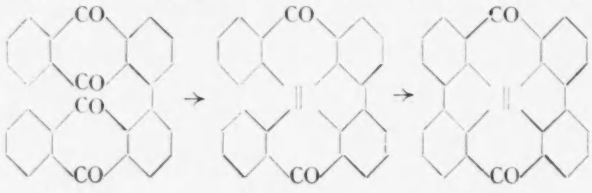
A resolution asking the executive to take steps to draw up a new national agreement in the dyeing and cleaning industry resulted in an equal vote of 28 each. A card vote showed a decision against. It was explained that although the resolution was defeated it would not prevent the executive from furthering the position by trying to get an all-in agreement.

The conference carried a resolution expressing the opinion that a strong effort thoroughly to organise the dyeing and cleaning industry throughout Scotland and England should be the immediate concern of the executive council.

Dyes and their Application : Helianthrone Dyes

By L. J. HOOLEY

ALTHOUGH the helianthrone derivatives have been known nearly as long as the idanthrones, they have not so far proved of importance. Discovery arose out of an observation by Scholl when preparing flavanthrone from 2 : 2'-dimethyl-1 : 1'-dianthraquinonyl *via* 2 : 2'-diamino-dianthraquinonyl, and helianthrone itself was first synthesised from 1 : 1'-dianthraquinonyl by reduction. The most satisfactory method is with copper in concentrated sulphuric acid at 40-50° C., although a number of other agents are suitable.



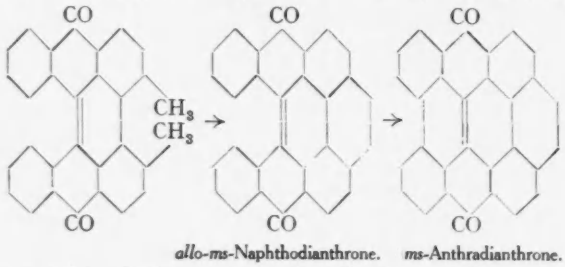
Helianthrone *ms*-Benzdianthrone *ms*-Napthodianthrone

It dyes cotton from the vat in yellow shades, and this suggested its name. As a dye it has the disadvantage of insufficient affinity, being removed from the fibre by treatment with hot alkaline hydrosulphite. Helianthrone itself was followed by a number of chloro-derivatives and other derivatives but these did not appear on the market. A number of dyes of undisclosed or unknown constitution have been obtained by the action of sulphuric acid and copper powder on anthraquinone derivatives, *e.g.*, Idanthrone Brown B, Caledon Brown B (B.P. 16,505 of 1907). Brown dyes obtained from dianthrone, naphthanthraquinone and other anthraquinone derivatives by aluminium chloride condensation may also be helianthrone derivatives. (G.P. 237751,241,631, Griesheim-Elektron).

Definite Compounds

To return to more definite compounds, it will be seen in the formula above that there is only required a link between the carbons in the 8 and 8' positions to form another benzene ring, and as would be expected this link can easily be made, the aluminium chloride bake or oxidation with manganese dioxide in sulphuric acid being effective. The systematic name for helianthrone is *ms*-benzdianthrone, the basis of which will be obvious. On the same principle the new body is *ms*-naphthodianthrone. It is an orange vat dye, but like helianthrone it has not found application in dyeing. It reduces with difficulty. Like helianthrone also a number of derivatives have been described but these have not been used either.

Within the last three or four years, however, developments of considerable interest have taken place. It has been found (B.P. 303,184, I.G.F.A.-G.) that when 2 : 2'-dimethylhelianthrone is treated with an alkaline condensing agent, oxidation takes place with formation of an additional benzene ring. The body is similar to but not isomeric with *ms*-naphthodianthrone, it is obviously still correctly spoken of as a *ms*-naphthodianthrone, but the prefix *allo-* is added. Halogenated and other derivatives have been obtained and as a class they are orange to red dyes with much improved tinctorial



allo-ms-Naphthodianthrone. *ms*-Anthradianthrone.

properties. Analogous to *ms*-benzdianthrone, *allo-ms*-naphthodianthrone forms another ring by linking the 8 and 8' carbon atoms, acid condensing agents, oxidising agents or sunlight being suitable.

The new body is *ms*-anthradianthrone, and is a singularly compact polycyclic compound having only twelve hydrogen atoms to thirty carbon ones. An alternative method which gives anthradianthrone is the alkali fusion of 2 : 2'-dimethyl *ms*-naphthodianthrone. A third method is from either of the two 2 : 2_1-dimethyl derivatives already mentioned, under more energetic alkali fusion conditions, and with an oxidising agent as well, is desired. (B.P. 315,033 *idem.*)

A large number of processes and products of halogenation of *allo-ms*-naphthodianthrone are described in B.P. 327,175 *idem.*). Treatment with chlorine or bromine in inorganic acids below 60° C. or in water, or other inorganic suspension media may be used. Under some halogenation conditions partial or complete formation of the anthradianthrone may take place. The products dye in orange-yellow to blue-red shades, and they may be purified by way of their oxonium salts or by the usual bleaching treatments. These halogenated derivatives may also serve as intermediates.

The Derivatives

The benz-, naphtho- and anthradianthrone derivatives may be nitrated (G.P. Applic., I. 37,140 *idem.*) and the resulting nitro-derivatives wholly or partly replaced by other groups such as chloro-, bromo-, ido-, cyano-, hydroxyl, amino- and alkyloxy- groups. Nitroanthradianthrone vats to give a blue dyeing. The hydrocarbon *allo-ms*-naphthodianthrone has also been prepared (by reduction of the quinone with hydriodic acid and phosphorus) and this has been nitrated. These derivatives appear primarily of interest as intermediates. In B.P. 327,702 (*idem.*) there are obtained iodo-anthradianthrones.

The various halogen and other simply substituted derivatives serve as starting materials for a series of more complex vat dyes. Thus, in G.P. Applic. I. 30,103 (*idem.*) halogenated *allo-ms*-naphthodianthrones are condensed with nitrogen compounds, particularly aminoanthraquinones. The dibromo-derivative gives in this way, with 1-aminoanthraquinone, a dark navy blue. *Ms*-naphthodianthrone derivatives behave similarly. (G.P. Applic. I. 30,064, *idem.*), red-violet, brown-violet, bordeaux and olive-green shades being described. In G.P. Applic. I 30,156 (*idem.*), the idea is extended to *ms*-anthradianthrones, condensation being effected with similar nitrogen-containing substances.

Of interest also are the reds and pinks obtained by halobenating alkyloxy *alo-ms*-naphthodianthrones, negatively substituted *allo-ms*-naphthodianthrones being treated with alcohol, phenols or mercaptans, and then halogenated. (G.P. Applic., I. 31,590, *idem.*).

Dyestuffs and Dermatitis

Methods of Testing

IN a paper read before the London Section of the Society of Dyers and Colourists by Dr. H. E. Cox, which is now published in the May issue of the Journal of the Society, mention is made of the increased number of cases of dermatitis alleged to be caused by dyed garments, including furs, and of the frequent lack of adequate proof of the supposed irritant action of certain dyes and of the difficulties due to idiosyncrasy. A list is given of dyed and other pigments which have been investigated experimentally mainly by Weyl and Sachs, showing which were found to be nocuous and which were innocuous. Many of the reports in medical literature on this subject are vitiated by inadequate information on the identity of the dyestuffs concerned.

Appropriate methods of extraction for testing are considered and figures given showing the extent of reduction of certain azo dyes by natural wool or fur and of Bandrowski's base in acid solution. As a result it is suggested that the suspect material should be extracted at blood heat with normal saline containing 0.1 per cent. of acetic acid and not with a mineral acid. It is suggested that the substances in question are only to be considered harmful when present in the free state in appreciable quantity and so transferable to the skin of a wearer.

Metallurgical Section

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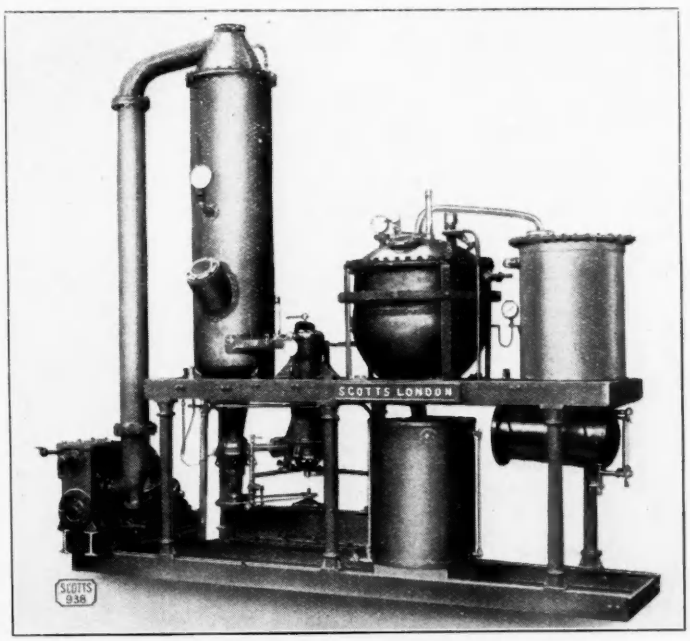

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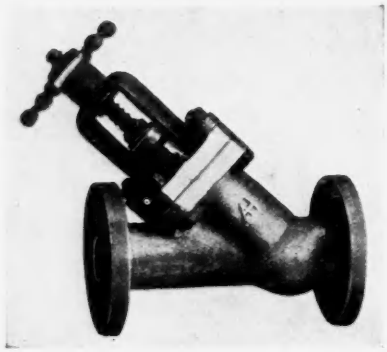

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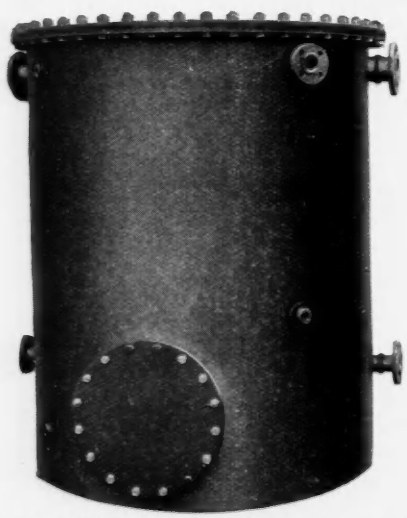

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Monthly Metallurgical Section

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Non-Metallic Inclusions in Steel

By J. C. Booth

There are two classes of inclusion which command the attention of those concerned with steel—oxides and sulphides—the oxides, which include the silicates, being probably the most important. This paper was read before the North Western Section of the Institute of Fuel at their meeting at Manchester on December 9.

THE majority of inclusions found in ordinary commercial carbon steels are the products of deoxidation, which process is essential in order to produce suitable steel for a given purpose. Oxygen reaches the basic open hearth bath by (1) oxidation of the scrap during the melting period, the smaller the scrap the greater the amount of oxide formed, (2) the decomposition of the limestone, (3) oxidation by furnace gases, and (4) direct addition of oxide during the refining period. In a well-designed charge the oxide resulting from the scrap is used up in eliminating most of the silicon and some of the carbon and phosphorus during the early stages of the heat, the silicon and phosphorus uniting with lime to form a slag which affords a measure of protection against oxidation by furnace gases. In order to bring the working period of the heat within commercial bounds it is necessary to activate the slag by direct addition of oxide, that is, during the refining period the slag must be oxidising.

Oxygen is soluble in steel, probably in the form of FeO, to an extent dependent upon the temperature and the carbon content of the bath—the higher the temperature the higher the solubility for a given carbon content, whilst, for a given temperature, the higher the carbon content the lower will be the solubility. The amount of oxygen in the metal depends also on the amount of oxygen in the slag and follows the partition law. It thus happens that when the metal is ready to tap there is present in the metal an amount of oxygen which depends on the oxygen in the slag, the temperature of the metal and its carbon content. It is for this reason that in the making of good steel the oxygen in the slag is kept within predetermined limits.

De-oxidation

In order to eliminate this oxygen the metal must be deoxidised. The usual method is to deoxidise with ferro-silicon and ferro-manganese. When silicon is added to molten steel it reacts with oxygen to form silica, which fluxes with some ferrous oxide to form a ferrous silicate. The type of silicate formed depends upon the ferrous oxide present in the steel before deoxidation and the amount of silicon added. Herty divides these into three classes: (1) those containing free FeO in a matrix of FeO-Fayalite eutectic and Fayalite in a matrix of FeO-Fayalite eutectic, (2) those containing dark spheres in a Fayalite-SiO_2 matrix, and (3) very dark glassy spheres of nearly pure silica. These types of inclusion correspond to different degrees of deoxidation.

When ferro-manganese is added to liquid steel the manganese reacts with the ferrous oxide to form manganous oxide which, provided the solubility of manganous oxide in the metal is not exceeded, will dissolve in the metal. On solidification the metal will contain inclusions in the form of grey globules consisting of a solid solution of iron and manganese oxide. Manganese is incapable of completely deoxidising steel and if a killed steel is required silicon must be added in addition to the manganese; this, under suitable conditions, produces iron-manganese silicate.

With low carbon steels, owing to the amount of oxygen present prior to deoxidation, it is almost impossible to kill the steel and keep it clean. It is for this reason that deep drawing sheet steel is only semi-killed, that is, a certain amount of oxygen is left in the metal, or not even killed at all, as is the case with rimming steel. In order to produce killed steel as free from inclusions as possible, since these are present in the deoxidised molten metal, it is essential that they be given opportunity to coalesce, and gather into larger mass, because the rate of elimination depends upon the particle size—the larger the particle the quicker will it be removed from the metal.

Silica is quite solid at steel-making temperatures, as also are iron silicates containing over 60 per cent. silica. Manganous oxide is also solid at steel-making temperatures, but since this forms a continuous series of solid solutions with ferrous oxide it is very unlikely that pure manganous oxide exists in steel. A consideration of these facts will show that the injudicious use of ferro-silicon for deoxidising will result in a dirty steel even though ferro-manganese is added afterwards.

Effect of Inclusions on Physical Properties

There is very little definite information regarding the effect of inclusions on the physical properties of steel. It is generally believed that metal high in oxygen is difficult to forge. This, as shown by Herty, depends to some extent on other factors. For instance, he found that steel, with 1 per cent. FeO, forged readily if the sulphur was low. He also found that steels containing ferrous silicate high in ferrous oxide forged well; this is probably due to the fact that at forging temperatures these silicates are semi-fluid and plastic. Steel containing silicates with a high silica content are difficult to forge because at this temperature they are quite hard, and may be considered as potential sources of cracks.

Kinzel and Crafts have studied the effect of inclusions on the impact strength, but they do not differentiate between the various types, which is very important. In order to explain why some comparatively dirty steels gave superior impact figures they introduce a vague term called "body." I have in mind a heat-treated nickel-chrome steel which gave 75 ft. lb. on the Izod machine which on examination showed a well-defined pipe. With regard to tensile strength the same capriciousness seems to obtain. A great deal depends on the type, size and distribution of the inclusions and the manner of testing. Undoubtedly the experimental difficulties are great. It would be wrong to say that, in all cases, inclusions are harmful. Free-cutting laminated spring steel and Aston metal are examples where inclusion material is intentionally introduced.

A New Iron of German Origin

A NEW special iron, called "migra iron" (abbreviated from micrographite) to mark its fine-fractured structure and its fine-grained graphite, is being manufactured by the Vereinigte Stahlwerke, Germany, and also, in a form rich in phosphorus at Gelsenkirchen-Schalke, and in a form poor in phosphorus, at the Frieidrich-Wilhelm smelter in Muhlheim. Professor Piwowarsky, of Aachen, reported on the use of this new iron for foundry purposes at the Haupversammlung des Vereins Deutscher Eisengiessereien, Berlin. The difficulty in its manufacture lies in a very definite intermediate-heat treatment of the crude liquid iron according to the principles of superheating melts. The total carbon and graphite content of the migra iron so obtained is lower, whereas the bending strength and Brinell hardness are higher.

Vanadium from Zinc Ores

A Recovery Process in Use in Northern Rhodesia

THE recovery of vanadic pentoxide from zinc-iron vanadate is now being carried out on a commercial scale at the works of the Rhodesia Broken Hill Development Co., Ltd., in Northern Rhodesia, where the zinc-lead deposits contain vanadium in the form of a hydrated vanadate of lead and zinc and a chloro-vanadate of lead.

In the extraction of the zinc from these ores for electrolytic recovery, the vanadium is dissolved and its recovery is effected in the form of fused vanadium pentoxide. The vanadium is first precipitated from the zinc solution as a zinc or iron vanadate, or as a mixture which is subjected to re-treatment for the recovery of the vanadium in a purified form. The precipitate is removed from the solution by filtration in filter presses, the filtrate being returned to the zinc circuit, and the cake is washed with water to remove most of the soluble zinc. When washing is completed, the precipitate is discharged from the press and transported to a storage bin, from whence it is fed, together with water, to a grinding mill, where the cake is converted to slurry. The mill discharge is then pumped to storage tanks, in which agitation is maintained, and from which pulp is withdrawn as required for the first stage of purification, where the vanadium is dissolved in caustic soda. The latter reagent is prepared on the plant by reaction between lime and soda carbonate, and is produced as a caustic liquor containing at least 75 per cent. NaOH.

Precipitation of the Vanadic Hydrate

Proportions of vanadium slurry and caustic solution, depending on their relative density and strength, are mixed in an iron tank provided with agitation and steam heating. The temperature is then raised to nearly 100° C. and agitation is continued until the whole of the vanadium dissolves as sodium vanadate, whilst the zinc, iron, etc., remain insoluble as hydroxides. The pulp is then pumped to a filter press and the purified solution passed on to the next stage of treatment. The residue in the press is washed with water and is discharged and transported to a pug-mixing machine, which delivers it to an agitation tank, where the residue is suspended in zinc solution and pumped back to the zinc circuit for recovery of that metal and utilisation of its basicity for further precipitation purposes. The sodium vanadate solution is then sent to the vanadic hydrate precipitation tanks, where sulphuric acid is added until free acidity is about 0.5 per cent. H_2SO_4, and by the introduction of steam the temperature is raised to a maximum of about 95° C.

The solution may be heated prior to addition of acid or acid may be added during heating, whichever of the two methods is most convenient. Acid is also added from time to time as may be necessary to maintain at least 0.5 per cent. excess, though an excess up to 25 per cent. can be added without detriment. As the temperature rises, the vanadic hydrate precipitates, maximum precipitation being obtained at boiling point. Excess of acid generally lowers the precipitation efficiency but may give a purer product; excess acid present during the precipitation period is therefore regulated at the amount required to produce maximum precipitation at the desired purity. The residual impurities in the solution remain dissolved and the precipitated vanadic hydrate is then practically pure.

Grade of Oxide Produced

When precipitation is completed the pulp is filter pressed in a wood-framed press, and the filtrate is either run to waste or retreated for its vanadium content, if this is still appreciable. Such re-treatment may take the form of returning the filtrate to the zinc circuit at a suitable point, if the content of other soluble salts permits. The vanadic hydrate precipitate in the press is washed until all soluble salts are removed, the wash being used in a counter-current manner and the strong wash returned to the precipitation circuit, after which the cake is discharged to a bin and then fed to a gas-fired melting furnace. Here the moisture and combined water are first drawn off, and the dehydrated material is then fused. The fused oxide discharges more or less continually into moulds, from which it is discharged, crushed and bagged. The grade of oxide produced is at least 90 per cent. V_2O_5, and may be 95 per cent. or more.

Organic Flotation Reagents

Increasing Demand for Blast Furnace Oils

THREE main methods predominate in the United States for the separation and concentration of ores—gravity, flotation, and combined gravity and flotation. Reports indicate that in 1929 over 96 per cent. of the ores were treated by the last two methods, both of which require the use of chemical flotation reagents. These chemicals are classified roughly as frothers, collectors, activators, regulators and depressors.

Frothing and collecting reagents are almost exclusively organic chemicals. Their total consumption in the United States in 1929 amounted to 9,283,005 and 8,269,546 lb. respectively. Frothers, utilised to make air bubbles of a more permanent nature, include cresylic acid, pine oil, ortho-toluidin, xylidin, aniline, ethyl alcohol, butanol and aldol. While frothers provide an abundance of stable bubbles, collectors or promoters, however, influence very markedly the capacity of these bubbles to carry such particles. For the last few years xanthates have proved the most popular collecting reagents, followed by coal-tar creosote and dicresol-dithiophosphoric acid and its salts. Other distillate tars and oils, as well as synthetic organic chemicals, are consumed in these processes.

The following table shows certain chemical flotation reagents consumed in the treatment of 65,404,827 tons of ores by 268 plants in the United States in 1929:—

	Total consumption lb.	lb. per ton of ore.
Pine oils	5,112,776	0.112
Cresylic acid	4,137,544	0.156
Ethyl xanthate	4,107,056	0.107
Butyl xanthate	194,252	0.054
Amyl xanthate	283,270	0.026
Coal-tar creosote	1,416,044	0.137
Dicresol-dithiophosphoric acid	610,983	0.045
Blast-furnace oils	590,614	0.069
Petroleum products	173,502	0.041
Thiocarbanilid	154,405	0.033
Wood-tar creosotes	120,010	0.028
Orthotoluidin	32,685	0.064
Coal tar	30,075	0.079
Alpha-napthylamine	23,471	0.104
Water-gas tar, oils	11,676	0.783
Benzyl mercaptan	650	0.021

In 1928 the total consumption of blast furnace oils was only 141,075 lb. or 0.013 lb. per ton of ore treated. The consumption of coal tar creosote, on the other hand, has decreased from 2,252,065 lb. in 1928, or 0.303 lb. per ton of ore.

Although reagent chemicals for ore flotation are not listed separately as such in United States official export statistics, some export trade is being developed with mineral-producing countries. In 1929 the United States supplied Canada with 135,036 lb. of "xanthates, cresylic acid, and compounds of cresylic acid, used in the process of concentrating ores, metals, and minerals," valued at $8,748, of a total importation of 341,630 lb., worth $22,650. Although there was a marked increase in total shipments of these commodities received by Canada in 1930, the American share fell to 1,142 lb., valued at $185. Germany and the United Kingdom are other sources of supply.

Totals for domestic production in 1930 of certain materials, used in part for ore flotation, are as follows:—Xanthates, 5,458,306 lb., ortho-toluidin, 1,240,980 lb., thio-carbanilide, 658,461 lb., xylidin and salt, 183,575 lb., cresylic acid (refined), 17,305,308 lb., coal-tar creosote, 122,571,822 gallons, coal tar, 602,485,929 gallons. Certain products such as aldol, alpha-naphthlyamine, and benzyl mercaptain are manufactured by only a few firms and production figures cannot be published without disclosing individual operations.

Copper Agreement

IT is announced that the representatives of the Belgian copper interests at the recent conference in New York have notified the representatives of Rhokana Corporation, Ltd., that they now accept the proposals with regard to curtailment of production made to them in New York to which the agreement of the other principal producers represented at the conference had already been obtained.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

New Standard of Purity for Mercury

THE effect of loose metal impurities on the properties of mercury intended for use in the making of dental amalgams has recently been studied by the United States Bureau of Mines. They have found that the presence of only 0.001 per cent. of such metals as zinc, tin, lead, cadmium, bismuth, copper, magnesium or antimony so changed the appearance of the mercury that anyone observing the metal would believe it to be badly contaminated. The changed properties of mercury caused by base metal contamination have been recorded before, but the fact that the appearance of the mercury may be used as a test for base metal contamination has never been emphasised. Contamination of mercury with silver or gold could not be detected by the changed appearance of the mercury. When either of these metals was present to the extent of 0.1 per cent. there still was no marked change in the appearance of the mercury. These observations indicate that mercury that is clean and bright and has the characteristic property of not wetting a glass surface cannot contain more than very minute amounts of base metal.

The Canadian Nickel Industry

THE importance of the Canadian nickel industry is now being brought to the attention of the public by an interesting display in the windows of the Ontario Government Office in London. This exhibit is planned to illustrate the wide ramifications of the Ontario nickel industry, demonstrating the extent to which so many manufacturing industries in this country are now dependent upon nickel as well as upon monel metal—one of the most important of the nickel-copper alloys derived from the Sudbury ore. In this rich mining district of Northern Ontario, nine-tenths of the world's supply of nickel is now being mined. Of no other base metal is the world's known commercial supply so largely concentrated at one point of the earth's crust. In one mine alone (the Frood) there has been discovered already sufficient nickel for the supply of the world's requirements for many years, calculated at the recent rates of consumption. Another mine about 10 miles away has been supplying about half of the world's requirements for most of the present century, and is very far from exhaustion. The first authentic discovery of nickel in the Sudbury district was recorded by the Dominion Geological Survey as far back as 1853, when Alexander Murray had his attention called to the strong deviation of the compass near what is now the famous Creighton mine. The first discovery of ore in place was made in a rock cut on the main line of the Canadian Pacific Railway near what was later known as the Murray mine. News of the discovery started active prospecting, and among the men who came into the field were many whose names have become prominent in the later history of the industry: Thomas Frood, James Stobie, Renaldo McConnell, F. C. Crean, J. H. Metcalfe, Thomas Baycroft, W. B. McAllister, E. Y. Eyre and William McVittie.

War-time Developments

BETWEEN 1884 and 1886, this group of prospectors located all of the now known large deposits, and in addition they traced the occurrence fairly completely around the rim or border of what has since become famous as the Sudbury Basin. In 1886 public attention was drawn to the new discoveries, particularly in connection with their association with the fortunes of the Canadian Pacific Railway project then under construction. At the same time, the British Government was made acquainted with the qualities of Canadian nickel. The output of nickel was then about 1,000 tons per annum, and up to 1903 it was used chiefly for nickel-silver, coinage and nickel-plating. Experiments made at a Glasgow steel works were the beginning of a new era of remarkable progress, for there arose a constantly increasing demand for nickel in armour plate, and nickel steel and ordnance, further expanded by the active naval competition between Great Britain and Germany. The demand continued to increase throughout the War period, and during the continuance of active hostilities the particular problem faced by the nickel industry was that of producing sufficient metal to satisfy the insatiable demand, the entire production being conserved for the Allies till the end of the War. Following the decisions of the Washington Naval Conference in 1921, however, the industry suffered a temporary set-back, and the whole attention of the companies concerned was concentrated on the maintenance of the remaining markets and the development of new ones. The success of their efforts will be readily grasped by consideration of the fact that production is now on a par with the peak years 1916-1917. The majority of products into which nickel goes to-day are engineering materials. The need, therefore, for competent research in promoting industrial uses of nickel products has been recognised, and a special department of engineering development and research has been created. It is a function of this department to secure the necessary engineering information on all metal products and to bring that information to bear effectively on every potential user.

Vanadium Steel from Sponge Iron

KUJI sponge iron, which is made by reducing titaniferous iron sand with "semi-coke" in a rotating furnace contains 50 per cent. metallic Fe, 30 per cent. FeO, 15 per cent. TiO_2 and 0.2 per cent. V, and so cannot be profitably used as scrap in steel making. Steel obtained from this sponge iron by ordinary methods of working in an electric furnace contain a considerable amount of phosphorus, and in order to obtain a vanadium steel low in phosphorus, it has been necessary to devise modifications of the method of steel making, which are outlined in a recent issue of *Metal Progress*. It is well known that although vanadium can be reduced by using a strong reducing slag, the prosphorus is also reduced and enters the steel. On the other hand, phosphorus is more reducible than vanadium at operating temperatures, and this difference has been utilised for obtaining a marketable low phosphorus vanadium steel. In this process a reducing slag is first made up in an electric furnace, the temperature being raised sufficiently so that a greater portion of phosphorus comes into the steel, while the most of the vanadium is contained in the slag, which is skimmed off and preserved in a ladle. Next, iron ore and lime are added to the furnace to make the slag oxidising and thus the phosphorus in the steel is largely removed. Finally, the phosphorus slag is replaced by the first slag; by adding coke into the furnace this slag is now made strongly reducing. Thus the vanadium in the slag goes into the steel, and a vanadium steel containing a low phosphorus is obtained. This steel contains 0.035 per cent. P, 0.10 to 0.15 per cent. V, and 0.10 to 0.15 per cent. Ti; it possesses good mechanical properties when properly heat treated.

Electro-deposited Nickel

THE influence of acidity of the electrolyte on the structure and hardness of electrodeposited nickel has recently been investigated by D. J. MacNaughton and R. A. F. Hammond (*Trans. Faraday Soc.*, October, 1931, pp. 633). In order to obtain further data concerning the influence of the conditions of deposition on the structure and hardness of electro-deposited nickel, deposits of this metal were prepared from three types of solution, in which the acidity has been varied over a wide range; the solutions were typical of those widely used in commercial practice. Examination of the Brinell hardness of the deposits and of their microstructure revealed that in all the solutions used the acidity exerts a very marked influence upon the hardness and structure of the deposit. In general, a low acidity (high pH) favours the formation of hard deposits having a finely crystalline structure, while a high acidity (low pH) favours the formations of soft deposits having a larger grain size. The hardness of the deposit was not found to increase uniformly as the pH was raised from the lowest to the highest value employed. In each solution there was a critical pH above which a rapid rise in hardness with pH took place, and below which only a small change in hardness with pH occurred. For the production of hard deposits the best results are obtained from a nickel sulphate solution buffered with ammonium salts, while for soft deposits made at a pH below the critical pH, nickel sulphate solu-

tions containing boric acid or sodium fluoride plus boric acid appear to be more suitable. The consideration of the bearing of the results on various views which have been advanced to explain the relationship between conditions of deposition and structure of deposit leads to the conclusion that (1) current density and cathode potential remain practically constant, whether hard or soft deposits are being produced, indicating that the structure of the deposit cannot be related directly to the intensity of the electric field of force at the cathode face which, it has been suggested, may control the crystal size of a deposit; (2) the view that the properties of nickel deposits are largely determined by an effect on crystal growth caused by the hydrogen discharged with the nickel only receives partial support; (3) the results give confirmation of the view that the structure of a nickel deposit may be greatly influenced by the co-deposition of nickel hydroxide or a basic salt.

Informal Agreement to Limit Copper Output

THE voluntary curtailment of the production of copper throughout the world came into effect this week, although no formal agreement has been drawn up and there is no authority to enforce the reduced rate. Output, according to the Copper Institute (New York) will be limited to 26½ per cent. of the estimated production capacity. More than 90 per cent. of the world's production was represented in the discussions. It was the view of producers that curtailment should cease when the total stocks of copper above-ground shall not equal in the aggregate the total deliveries in the preceding four months. Moreover, if the price of copper shall exceed for a period of 15 days the price of 12 cents. a pound, Connecticut Valley delivery, the curtailment shall cease, no matter what the existing stocks may be. Should the total stocks not have been reduced by December 21, 1932, so as to equal the total deliveries of the preceding four months, any company may resume operations as it sees fit on giving 30 days' notice. The new Rhodesian production and several Canadian and South American mines not hitherto members, are expected to join Copper Exporters Incorporated on condition of a modification of certain of its rules. The 26½ per cent. reduction voluntarily agreed to in New York by producers controlling 90 per cent. of the world's copper output will still allow the Union Miniére (Belgium), however, to produce about 82,000 tons annually. Of these 82,000 tons authorised as from January 1, 1932, 30,000 will remain in stock in the African mines until required.

Precautions in Heat Treatment

THE high-speed steel generally used in the United States is known as the 18-4-1, and commonly contains 0.60 to 0.75 per cent. carbon, 17.00 to 18.50 per cent. tungsten, 3.25 to 4.50 per cent. chromium, and 0.60 to 1.25 per cent. vanadium. Some of the so-called super-high-speed steels also contain 1.50 to 3.50 per cent. cobalt. In carbon steels the principal object of forging is to reduce cross-section; this operation also breaks up the coarse crystals and yields a fine-grained structure. The coarse cast structure of carbon steel may also be refined by heat treatment. In high-speed steel, however, this is not the case. As cast, this material has a coarse heterogeneous structure, whilst embedded in the matrix are hard and brittle carbides of iron, tungsten, chromium and vanadium. Very commonly these complex carbides take the form of cells which resist disintegration or solution with any known heat treatment. Hence forging is applied to the cellular cast structure and if brittleness and possible failure are to be avoided in the finished tool it must be done thoroughly. The annealing of high-speed steels after forging has two objects—first, to soften the material so that it can be machined, and secondly, to make the structure uniform. In this type of steel, the tungsten is of greatest importance in retarding the tempering of the martensite. Together with chromium and vanadium, tungsten also forms double carbides with iron and carbon which are intensely hard and which retain their hardness even if heated to a red heat.

Use of Metals at Elevated Temperatures

IN a recent paper read before the American Society for Testing Materials, R. W. Bailey and J. H. S. Dickenson have listed the desirable attributes of alloys for high temperature service. Combination of all the attributes is unobtainable and the choice must be made on the basis of the most important requisites. Nickel does not, in itself, improve high temperature load carrying ability of steel. Of low alloys steel 1 per cent. Cr. 0.25 per cent. V is good to 750° F. One of 2.5 per cent. Ni, 0.7 per cent. Cr, 0.3 per cent. Mo is good to 930° F. Above these temperatures, the steels have high creep. Plain 0.15 per cent. C, 0.35 per cent. Mo steel has remarkably high creep resistance to 1,020° F. and its extended use is anticipated. Increase in Cr increases creep resistance and adds corrosion resistance. Austenitic Ni Cr steels have attractive creep resistance to 1,110° F., but are prone to intercrystalline fissuring if heated in the range 930-1,560° F. Automobile engine valves for heavy duty are trending toward use of austenitic Ni-Cr steel in England. Embrittlement by many cycles of heating to 730° or 840° F. occurs in heat-treated mild steel. Nickel makes it worse. Sufficient molybdenum cures it. No steel which suffers from this effect should be used for parts stressed at high temperatures. High silicon cast iron with fine graphite resists growth and oxidation to 1,650° F. Mild steel scales badly above 930° F. A 7½ per cent. Cr steel, with carbon below 0,12 per cent. and perhaps with silicon up to 1 per cent. has good scaling resistance to 1,290° F. above which the heat-resistant steels and alloys are required. The cast 65-20-15 Ni-Cr-Fe alloys must be cast sound for good service. Cast Ni-Cr-Al alloys are suggested for use to 2,000° F. in sulphur gases. A plain 30 per cent. Cr steel is very resistant to oxidation and to sulphur, but is brittle cold.

The Father of Chromium Plating

Although the literature of chromium plating dates back to nearly 100 years ago, the solution of the difficulties in the successful application of a commercially feasible process giving really satisfactory results is due primarily to Professor Colin Fink, and dates back no further than the year 1925, when his application for a patent was first filed, to be granted on April 20 of the year following. That patent (U.S. No. 1,581,188) which may be regarded as the master patent controlling modern chromium plating practice has been the subject of an important law suit in the United States and judgment was given recently, in the action brought by the United Chromium, Incorporated, against the International Silver Co., for its infringement, upholding it. That judgment has been reported in full in the columns of our contemporary *American Metal Market*, for November 19 and 20. It is one of very great importance to the plating trades, and apart from its commercial and technical interest it is refreshingly downright and lucid, and comprises a philosophical review of what constitutes an invention, and entitles an inventor to patent protection which is a pleasant revelation of an aspect of the American Courts hardly known on this side of the Atlantic where the rough and tumble methods familiar to many of us through the "movies" convey a false impression of the administration of American law.

Grades of Inventions

The judgment was delivered by Judge Thomas, at the District Court, Connecticut. The Judge quoted, in that judgment, the whole of the patent preamble and the detailed clauses of the specification which need not be recapitulated here. Its main point was insistence on securing and maintaining the correct ratio between the chromic acid and the catalyst radical CrO_3/SO_4. This had been ascertained by Professor Fink, after laborious researches. The patent thus covered what, in the judgment, was referred to as "two series of inventions." There was first the discovery of the difficulties (or successful chromium plating) and second, the discovery of means to overcome those difficulties. "Prior to Professor Fink," said Judge Thomas, "there was no really practical, satisfactory, successful, and commercial chromium plate. The commercial success of Professor Fink's method consists not only in producing chromium-plated articles of every kind and description which meets in all fields the requirements of the trade and the public in general, but also because the plating process is made continuous which obviates waste in the electrolyte in the steady plating operation day in and day out. Both these features were unknown prior to the patentee's invention. The plating on certain pieces of metal were at times nearly a success, and often they were partly a failure, or a complete failure." Professor Fink is to be complimented on having his patent upheld.

The Iron and Steel Industry

World Conditions During October

ACCORDING to the *Statistical Bulletin* issued by the National Federation of Iron and Steel Manufacturers, production figures for the United Kingdom during October show a welcome break in the downward trend of production. The production of pig iron rose from 248,200 tons in September to 284,200 tons in October, while the number of furnaces in operation increased by four to 66. Pig iron production, however, was only 70 per cent. of the rate in October, 1930. Steel production rose from 400,500 tons in September to 457,400 tons in October. Imports of iron and steel which had been 220,300 tons in September rose to 275,700 tons in October, the chief items responsible for the increase being pig iron and billets, blooms and slabs. Exports of iron and steel rose slightly from the extremely low level of September to 158,800 tons in October, the items responsible for the increase being mainly tinplates and galvanised sheets.

So far as Germany is concerned, a fair number of orders are reported to have been booked from South America and the Far Eastern markets. Keen competition was experienced from Belgian and Luxemburg producers for the volume of business available, particularly in the Northern European markets.

In a speech delivered before a gathering of leading industrialists in America, Dr. Fritz Thyssen, the German Ironmaster, stated that, as the consumption of steel is a barometer of industrial growth or decline, it is not surprising that in the present world depression the steel industry in all countries is depressed. The extent of the depression, however, differs in different countries indicating that other factors, peculiar to each country, have been operating in addition to the general world factors. As regards Germany, the post-war development of the steel industry has been conditioned, *inter alia*, by the repercussions of the Treaty of Versailles and, in particular, reparations: the influence exerted on general social and industrial policy by the trade unions and the political parties associated with them: and the excessive expansion of credit which took place in 1924-29.

France and Belgium

In view of the depressed state of trade, French steel producers are paying increased attention to the question of reducing costs. Certain works are damping down blast furnaces and increasing the proportion of scrap in the charge for the steel furnace which, in view of the present low price of scrap, is more economical. In other works, however, the blast furnaces are being kept in full operation owing to the fact that the surplus gases are used in subsequent operations. In the Longwy Basin activity has up to the present been fairly well maintained in the iron and steel works, but some anxiety is felt regarding the future as it is feared that the continued shortage of orders will render the dismissal of many workers inevitable.

Owing to the lack of a controlling authority enforcing a uniform policy, the Belgian iron and steel industry has not adapted itself to the difficult conditions of the past twelve months. At the Athus plant three out of four furnaces have been kept in blast and various improvements have been effected during the past year. At Grivegnée the plate mill has been operating below capacity owing to the unfavourable demand. In his speech at the annual meeting the chairman stated that there is little ground for optimism but the reconstruction of the international agreement should lead to an improvement in the position. With reference to the position in the United Kingdom he expressed the hope that any duties which might be imposed would not be fixed at too high a level.

United States

During October operations in the steel industry rose to 31 per cent. of productive capacity, and it is expected that they will show a further improvement. The improved sentiment of the market also found more tangible support in the heavier structural steel awards. At the Autumn meeting of the American Iron and Steel Institute, Mr. J. A. Farrell, President of the United States Steel Corporation, emphasised the fact that America had the richest home market in the world with an immense consuming capacity and they must therefore discover means of increasing the consumer's demand. He condemned the reduction of prices to uneconomic and profitless levels in an endeavour to attract business which affords an artificial decline in commodity prices leading to a vicious circle of a lower buying power generally. Mr. C. M. Schwab, Chairman of the Bethlehem Steel Corporation, stated that they needed a better financial structure, a liquidation of extravagance in business, a reduction in the cost of Government, and a vigorous enterprising attitude on the part of industry.

Aqueous Solutions for Quenching Steel

THE more common steels are made soft by cooling slowly from temperatures approximating a "red heat," and are hardened by cooling rapidly from these same temperatures. Quenching, that is, cooling rapidly from a red heat by plunging metal into water or oil, is thus a fundamental process in heat treatments employed to develop enhanced properties. The precise structure and properties produced in quenching are controled by the speed at which the steel cools during the operation of water and oil which are the two most commonly used quenching media, water produces the more rapid cooling effect, giving a steel which is generally harder than one quenched in oil. Occasionally a water-quenched steel is too hard for the most satisfactory service, and in such cases a tempering treatment is frequently necessary, which consists of reheating the steel after quenching to relative low temperatures. In a recent investigation at the United States Bureau of Standards a study was made of aqueous solutions of ethylene glycol, glycerine, and sodium silicate to determine whether any of them offered promise as quenching media with characteristic cooling rates intermediate between water and oil. It was found that the solutions of glycol and of glycerine did not give results which were readily reproducible. Solutions of several concentrations of sodium silicate, however, proved to have quenching rates in the desired intermediate range and to give very consistent results. Some of these sodium silicate solutions must be treated with small additions of caustic soda in order to prevent changes in them when allowed to stand for several months. The effect of varying the concentration and the temperature of the sodium silicate solution was studied. It was concluded that sodium silicate solutions give promise of usefulness in the quenching of steels by providing solutions having graded cooling rates between those of water and of oil.

Birmingham Iron Trade

ACCORDING to the *Birmingham Post* steel makers are waiting anxiously for what the New Year will bring them in the shape of increased business and, particularly, in increased specifications against orders already on their books. In the small steel bar and semi-crude branches practically no business was transacted last week, nor was any change notified in prices, though of these there was no test. The holiday output of pig-iron will go into stock, but the position in that respect is much less embarrassing than it was before September brought with it a welcome stirring of demand. Throughout the last three months of the year consumption has been fairly regular, and furnacemen are well sold over the early weeks of the New Year at current rates, in some cases, indeed, until the end of the first quarter. Foundry iron is a somewhat meagre feature of the market, denoting the state of the Staffordshire iron industry.

Test on Steel for Turbines

AT the request of the British Electrical and Allied Industries Research Association an investigation was undertaken in the engineering department of the National Physical Laboratory as part of the general programme of work for the Committee of the Department of Scientific and Industrial Research appointed to supervise researches on the behaviour of materials at high temperatures. Engineering Research Special Report No. 17 (H.M. Stationery Office, price 9d. net) which has now been issued, contains the results of short-time tensile tests and creep tests carried out on a 0.30 per cent. carbon cast steel and a 0.39 per cent. carbon forged steel over a temperature range of approximately 400° C. to 650° C. Full tensile properties and the stresses corresponding to rates of creep down to 10^{-5} inch per inch per day have been determined. The results are given in numerous tables and curves.

Some Inventions of the Month

By our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Aluminium and its Alloys

IN a process described in Specification 355,792, dated May 22, 1930, by T. R. Haglund, of Stockholm, Sweden, for the manufacture of aluminium or aluminium alloys from alumina or materials containing alumina, an alloy containing tungsten is first produced by an electrothermic smelting with a reducing agent, e.g., carbon, and an addition of tungsten-containing substances with or without a flux, and aluminium or a high grade aluminium alloy is then recovered from the tungsten-containing alloy. In the production of aluminium from pure alumina this recovery is effected by distillation, preferably *in vacuo*, or by an electrolytic refining, and in the production of aluminium alloys, containing, e.g., iron, silicon, or titanium, present in impurities in the alumina, the recovery is by cooling and crystallisation or liquation. The tungsten-containing alloy has preferably an aluminium content of 50-90 per cent. The crystals of rich tungsten-aluminium alloy produced are separated from the liquid metal by contrifuging, pressing or other means and are returned wholly or in part to the smelting charge. The tungsten-free aluminium or aluminium alloy finally obtained may be further refined electrolytically.

Annealing Alloys

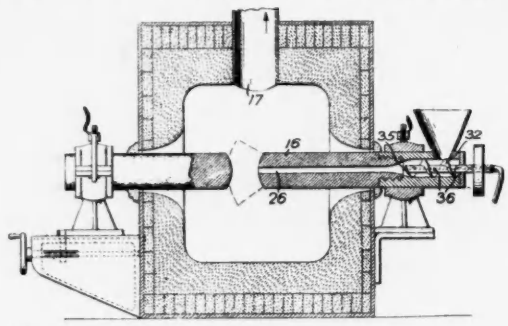
OBJECTS made from binary magnesium-manganese alloys containing about 0.5-2.5 per cent. of manganese have their resistance to corrosion increased by subjecting them to a heat treatment at above a minimum temperature which is about 400° C. for an alloy containing 0.5 per cent. to about 480° C. for an alloy containing 2.5 per cent. of manganese. Such heat treatment may be combined with hot working of the objects, but if working is carried out at a temperature below the minimum for the heat treatment it should be followed by a separate heat treatment. In any case the treatment may be followed by quenching. In an example a cast block of magnesium-manganese alloy containing 1.4 per cent. of manganese is rolled into sheets which are annealed for 48 hours at about 500° C. and allowed to cool in air. See Specification 355,920, International Convention date, August 23, 1929, by I. G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany.

Concentrating Copper Matte

IN the concentration of a copper matte by treatment with mineral acid to produce hydrogen for reducing part or all of the copper sulphide present, the matte is first rendered readily reactive toward the acid by increasing its content of free iron. A content of 2-12 per cent. of free iron is suitable for this purpose. Scrap iron may be added to the charge of a copper smelter or to the molten matte, or a mixture of finely divided matte and metallic iron may be used. Alternatively the iron may be produced in the matte by reduction of iron oxides present, e.g., by allowing molten matte to flow down a column containing glowing coke or by injecting coal, coke, hydrogen, carbon monoxide, producer gas, oil, oil gas or other reducing agent into the molten matte. See Specification 355,078, International Convention date, September 25, 1929, by United Verde Copper Co., of Clarkdale, Arizona, U.S.A.

Extracting and Refining Metals

AN application of the electric arc for extracting and refining iron and other metals and for the preparation of alloys is described in Specification 355,700, International Convention date, June 27, 1929, by Buffalo Electric Furnace Corporation, of Buffalo, New York, U.S.A. The intimately mixed powdered charge is continuously forced, e.g., by a worm conveyor 32, through the bore 26 of a conduit 16 terminating in or adjacent to the arc. Preferably, the conduit itself constitutes one of the electrodes. The intermediate portions of the conduit are located within the furnace and are maintained at such temperatures that the charge reacts and becomes fluid before it reaches the arc. The conveyor is formed with a bore 35, provided with openings 36, for the supply of a trace of gas in admixture with the charge. Such gas may be diverted from the gases leaving the furnace by the flue 17. Carbon monoxide produced during the reduction of iron ores in the arc may be thus mixed with the charge. Operating conditions for the production of ferromanganese from a



355,700

charge of iron, pyrolusite, carbon, and flux are specified in detail. Reference is also made to the reduction of complex ores containing nickel, the recovery of metals from fine dusts, and the production of ferrosilicon and of carbides of calcium, tungsten, etc.

Ferro-Phosphorus

A PROCESS for obtaining ferro-phosphorus free from or poor in silicon and having a high phosphorus content from ferro-phosphorus rich in silicon is described in Specification 355,610, International Convention date, December 17, 1929, by I. G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany. The material rich in silicon is mixed, preferably in a fluid condition, with tricalcium or other raw phosphate, preferably in about 50 per cent. excess, and is fused at 1,450°-1,600° C. in a non-oxidising atmosphere, e.g., in an electric furnace. The silicon reduces the phosphate to phosphorus which in part is incorporated in the ferro-phosphorus and in part escapes, while the silica produced forms a slag with the lime. The ferro-phosphorus obtained contains up to 30 per cent. of phosphorus.

Sintering Ores

A PROCESS for sintering carbonate or oxide ores described in Specification 355,564, International Convention date, November 16, 1929, by Metallges Akt.-Ges., of Frankfort-on-Main, Germany, Comprises a preliminary calcining of the ores, e.g., in a rotary kiln, shart furnace or blast apparatus, using just sufficient fuel to effect expulsion of the carbon dioxide and water, thereafter adding just sufficient fuel to effect the sintering, and finally sintering in a blast apparatus through which air is drawn or forced, e.g., a Dwight-Lloyd apparatus. An addition of fine grained clinker or blast furnace slag may be made to the charge to be sintered.

Zinc

THE removal of cadmium or cadmium and lead from zinciferous materials is effected by subjecting them in the form of a porous moist layer to a sintering operation in the presence of a chlorinating agent, under such conditions that no formation of strongly basic reaction products occurs, so that cadmium and lead chlorides are volatilised with substantially no volatilisation of zinc. Specified chlorinating agents are zinc chloride, hydrochloric acid, and a mixture of zinc sulphate, sulphuric or other acid and a chloride, e.g., of sodium, calcium or barium. In an example a partly roasted zinc concentrate is mixed to a suitable consistency with 5 per cent. of coal dust and 5 per cent. of zinc chloride in solution, and the mixture is ignited for a period which is about six times that required to ignite the surface of the charge. The discharged sinter should not be red hot. After collecting the volatilised fume in water the cadmium and lead in the solution may be replaced by zinc, and the zinc chloride so produced may be re-used in the sintering operation. See Specification 355,840, International Convention date, October 15, 1929, by New Jersey Zinc Co. of New York, assignees of H. M. Cyr and R. K. Waring, of Palmerton, Pennsylvania, U.S.A.

Monthly Metallurgical Section

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NOTICE.—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154 Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.*

The Removal of Bismuth from Lead

Use of Metallic Calcium and Calcium Alloys

The use of calcium for removing bismuth and other impurities from lead should provide an adequate and commercial demand for electro-chemically produced calcium which has hitherto received little attention from the manufacturer.

BISMUTH is commonly separated from lead by an electrolytic process, in which the lead bullion is used as the anode and pure lead is deposited at the cathode, leaving an anode sludge consisting principally of bismuth and other impurities. This process, however, is somewhat uneconomical when applied to lead containing less than 1 per cent. of bismuth. The problem of removing 0.1 to 0.35 per cent. of bismuth from de-silvered lead therefore becomes a matter of importance when large quantities of de-silverised lead are produced, as there is still a large demand for white lead as a paint component and specifications for lead for use in the corroding process usually limit the bismuth content to 0.05 per cent.

Effect of Alkaline Earth Metals

This problem of controlling bismuth to the extent required for producing "corroding lead" from ordinary lead bullion first received the attention of Dr. G. J. Kroll, of Luxemburg, who showed that alkali or alkaline-earth metals added to a non-ferrous alloy caused a dross to form in which certain constituents of the alloy were concentrated. Accordingly a patent was granted to Kroll in 1922, in which it is stated that the separation of bismuth from lead containing 0.2 per cent. Bi. resulted in the production of a purified lead carrying only 0.05 per cent. Bi. and a dross which carried 5 per cent. Bi. Ultimately, at the University of Michigan, experiments were taken in hand to determine the possible economy of such a process in relation to the Parkes' process of lead refining, where copper, silver, gold and most of the arsenic and antimony are removed by well established methods, whilst bismuth is not affected. In this investigation metallic calcium was introduced in various percentages at about 900° F., after which the lead was stirred and allowed to cool. During the cooling a dross was removed, similar in appearance to the zinc-silver dross of the Parkes' process, and as shown by Kroll, the bismuth content of the lead was reduced to slightly below 0.05 per cent., whilst the last traces of arsenic, antimony, silver and copper were completely scavenged. The amount of dross formed varied with the amount of impurities present, and contained up to 10 per cent. Bi. for lead with varying bismuth contents within the economic range of the process.

A portion of the calcium remains in the lead, depending partly on the excess of calcium introduced and partly upon upon the solubility of calcium in lead at the melting point of lead. Such residual calcium when present in amounts even less than 0.05 per cent., makes purified bullion very hard, but this hardness may be corrected by removing the calcium by blowing with steam, chlorine or other oxidising materials.

There is considerable evidence in the literature to the effect that calcium forms intermetallic compounds with most of the non-ferrous metals, the compounds so formed having melting points considerably higher than either of the constituent metals. Inter-metallic compounds of calcium are well established for lead, zinc, silver, magnesium, aluminium, mercury, cadmium and tin. There is contradictory evidence, however, with respect to bismuth which makes it difficult to state that the de-bismuthising action is due to compounds of bismuth forming in preference to those of lead.

The De-Bismuthising Process

The process may be considered as involving four steps:—(1) dissolving the reagent, (2) drossing and liquating, (3) removal of the residual calcium, and (4) treatment of the dross to remove calcium and recover the bismuth. Each of the first three operations involves only moderate processing charges per ton of bullion treated. The fourth step involves the treatment of the dross by means of the Betts electrolytic process. The amount of bismuth in the original bullion determines the amount of calcium required, and the amount of calcium required determines the amount of dross formed. Hence the cost of the first three operations is fixed and the cost of the fourth operation and of the calcium used increases with the bismuth content. For bismuth contents of 0.1 per cent., the cost of the calcium would be about equal to the cost of the operations or half the total cost. With double the bismuth content, the calcium could be used more efficiently, so only slightly more would be required and the total cost would be increased but slightly. Above the point of full efficiency the cost per ton of lead treated will increase, due to the larger amount of calcium consumed and to the larger amounts of dross formed. It is therefore likely that this process has its most economical application when used on lead having 0.5 per cent., or less, of bismuth and other removable impurities.

The commercial success of the process is dependent on four factors. The first is a means of making calcium metal or alloy cheaply; the second is a demand for bismuth in excess of that available from other processes, which implies the discovery of new uses dependent on the physical and chemical properties; the third is a shortage of lead of high purity, due to increased demand for pure lead; and the fourth would be a demand for lead of high purity and of greater hardness than ordinary pure lead. The last factor is the one most likely to be of immediate interest. Hard lead alloys containing considerable calcium, such as Friary metal, are well-known, but alloys containing 0.08 per cent. or less are not so well-known. Such alloys are being used experimentally for cable sheath, and similar purposes.

Production of Calcium Alloys

Metallic calcium for use in the process may be produced by the electrolysis of fused calcium chloride, a current efficiency of 60 per cent. being obtained at 25 volts, with low current density. The product obtained, however, is to a certain extent contaminated by gaseous and solid impurities and may be difficult to dissolve in the lead. Calcium alloys, on the other hand, can be produced by the electrolysis of fused calcium chloride using a molten lead cathode, graphite anode and an iron container. A considerable tonnage of such alloys has already been produced in certain parts of Europe, current being supplied at 10 volts and each cell operating at 2,000 amps., giving a production of about 1 lb. of calcium per hour in the form of a 4 per cent. alloy.

Microscopic Investigation of Ores and Metals

THE January issue of the *Canadian Mining and Metallurgical Bulletin*, which is published by the Canadian Institute of Mining and Metallurgy, contains a paper by F. F. Osborne, assistant professor of geology, McGill University, Montreal, entitled "Plane-Polarised Light in the Microscopic Investigation of Ores and Metals." This paper has been prepared for discussion at the annual general meeting of the Institute to be held at Montreal in April next.

National Metallurgical Problems

Research under the Auspices of the D.S.I.R.

METALLURGICAL research receives considerable attention in the Report of the Department of Scientific and Industrial Research for the year 1930-31, recently published by H.M. Stationery Office (price 3s. net). Work has been carried on throughout the year at the National Physical Laboratory upon the behaviour of materials at high temperature, in accordance with the programme recommended by the supervising committee of the Metallurgy Research Board. Investigations on the nature and mechanism of creep have also been continued, special testing units having been designed for the detection of very small rates of creep. At the laboratories of the British Non-Ferrous Metals Research Association an attempt is being made to discover new types of non-ferrous alloys suitable for service at high temperatures, the effect of additions of aluminium and silicon to 80: 20 cupro-nickel being at present under investigation. Striking improvement in heat-resistance has already been noted. In the research for new ferrous materials, carried out under the supervision of a joint committee of the National Federation of Iron and Steel Manufacturers and the British Electrical and Allied Industries Research Association, the first step has been to ascertain the mechanical, and in particular, the creep properties of steels containing only moderate additions of alloying elements.

Light Metals Research

Research has continued during the year at the National Physical Laboratory on aluminium alloys, and has been restarted on magnesium alloys, while some attention has also been paid to alloys of beryllium. In view of the immediate practical importance of the development of magnesium alloys, these alloys are being given special priority. A study of the age hardening and modification of aluminium alloys is also in progress, earlier work being repeated with the very pure aluminium that has recently become available. The work carried out under the supervision of the Minor Metals Research Committee has been almost entirely concerned with the production of pure beryllium, with the object of obtaining the metal in a ductile state, and while success has not as yet been attained, further methods of attack are being investigated. Further investigation is being made into the possibilities of beryllium as an alloying element.

Investigation has been begun into the effect of corrosive media on the behaviour of boiler steel when subjected to relatively severe plastic deformation. It has been found that when test specimens are bent at ordinary temperatures in alternate directions at intervals of 24 hours, continuous immersion in tap water reduces the number of bends before fracture by 30 to 40 per cent., as compared with air. Concentrated solutions of sodium chloride have a similar, but less severe effect. Besides their importance in the present problem, the results obtained may have some bearing on the operation of hydraulic machinery and such articles as anchor chains, where there is the possibility of severe stresses being applied intermittently to parts continually immersed in water. The prolonged loading tests at elevated temperatures in air on notched and on drilled mild steel test specimens have also been continued. This work is being extended to include tests at elevated temperatures on specimens totally immersed in corrosive media.

Electro-Deposition

The investigations on problems of electro-deposition in progress at the Research Department, Woolwich, and Sheffield University, have been continued throughout the year. Work at Woolwich on the cause and prevention of pitting in electro-deposited nickel has, in all essentials, been completed. Methods for avoiding the defect have been established and a comprehensive explanation of the various phenomena observed has been obtained. Progress has been made in a study of the chief factors which operate during deposition to determine the physical properties of nickel deposits. Some work has been carried out on the heat treatment of electro-deposited nickel. A further study of the best conditions for the deposition of nickel by the combined use of alternating and direct current has been in progress at Sheffield University, where experiments are being made in the use of "aged" solutions, which are found to be the most suitable for the production of thick nickel deposits.

British Non-Ferrous Metal Industries

Census of Production Statistics

THE results of the fourth Census of Production (1930) in iron and steel foundries in the non-ferrous metals and in the gold and silver refining trade, are summarised in a series of preliminary reports which have been published in the *Board of Trade Journal*. Iron and steel foundries were grouped as a separate industry for the first time. In the previous Census the production of iron and steel castings was distributed between various metal-working industries, the chief of which were the heavy iron and steel trades, which covered the smelting and rolling of iron and steel and the founding of heavy castings, and the light castings trade, which included machined products, such as appliances for heating, sanitary and ventilating purposes.

The returns for 1930, covering 767 establishments, give the following totals for the whole of Great Britain:—

Gross output	£27,524,000
Cost of materials used	£11,426,000
Net output	£15,938,000
Persons employed	84,819
Net output per person	£188

The gross output included 499,300 tons of pipes and fittings, valued at £5,268,000; 166,100 tons of cast-iron stoves, grates and ranges, valued at £6,497,000; 103,900 tons of railway material, valued at £985,000; and 251,400 tons of engineering castings, valued at £5,136,000.

Copper and Brass

The returns for the copper and brass (smelting, rolling and founding) trades cover 230 establishments employing more than ten persons. The total value of the production of the chief classes of copper and brass and manufactures thereof was returned at £21,115,000 for 1930, as compared with £23,240,000 for 1924. The figures for both years are inclusive of similar products returned by firms chiefly engaged in the smelting, rolling etc., of other non-ferrous metals, including similar products of those returned on schedules for the wire trades. The total sums so included are £3,227,000 for 1930 and £3,138,000 for 1924. In addition, the proceeds of sales of copper scrap were returned for 1930 at £145,000, and of brass scrap at £163,000. The following figures show the total cost of materials used:—

	1930.	1924.
Cost of materials used	£14,303,000	£16,716,000
Net output	£5,524,000	£6,128,000

The average number of persons employed by these firms was 23,200 in 1930, as compared with 26,478 in 1924. The net output for each person employed was £238 in 1930 and £231 in 1924.

Lead, Tin and Aluminium

Returns, covering 188 establishments, from firms chiefly engaged in the smelting, rolling and casting of lead, tin, zinc, aluminium, and other non-ferrous metals (except copper and brass and precious metals), give the following totals:—

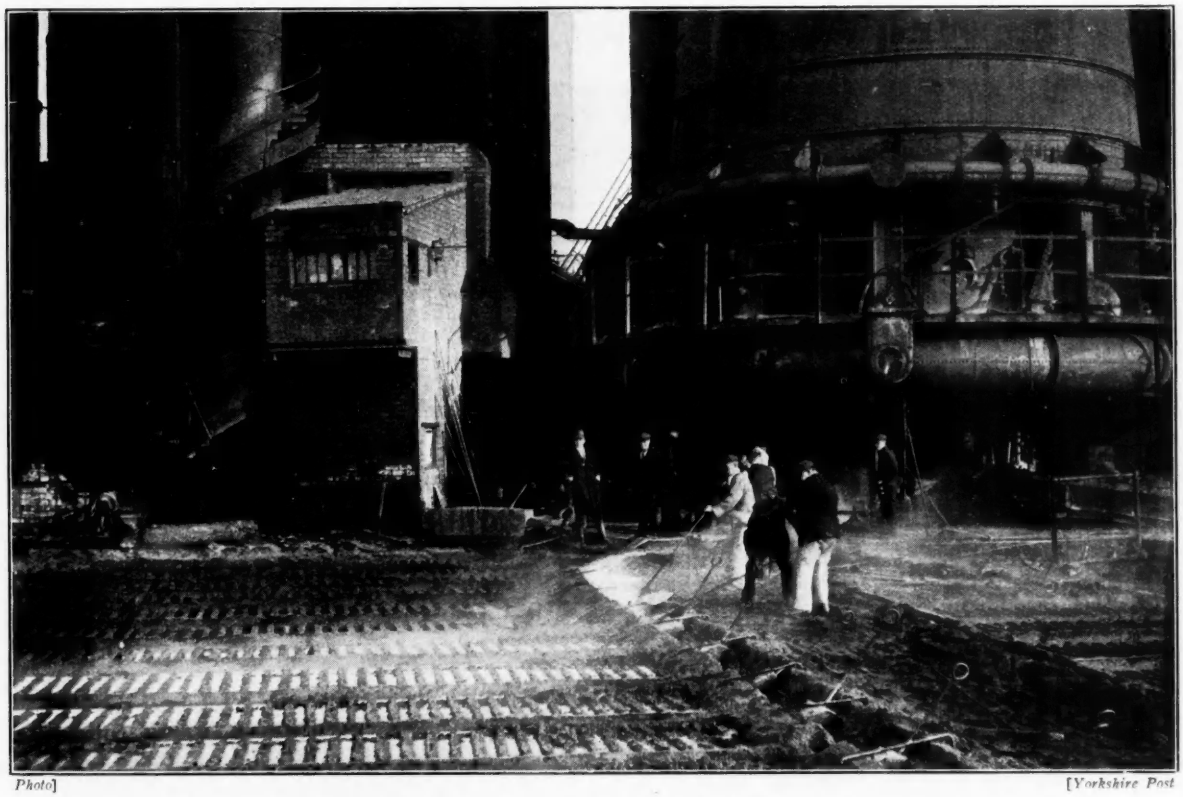
	1930.	1924.
Gross output	£24,052,000	£31,662,000
Cost of materials used	£18,851,000	£25,133,000
Net output	£5,738,000	£6,491,000
Persons employed	19,907	22,280
Net output per person	£288	£291

The gross output figure for 1930 includes £1,265,000 for the output of the specified products by copper and brass smelters and refiners of precious metals. The average selling values of lead products in 1930 were only about 60 per cent. of the prices ruling in 1924, of tin 72 per cent., of aluminium 79 per cent., and of zinc 52 per cent.

The returns from establishments engaged in the refining of gold, silver and other precious metals show:—

	1930.	1924.
Gross output	£39,713,000	£15,796,000
Cost of materials	£38,487,000	£14,801,000
Net output	£1,220,000	£985,000
Persons employed	2,245	1,778
Net output per person	£544	£554

The gross output figures for 1930 included £33,027,000 for refined gold and gold manufactures (as compared with £8,442,000 in 1924); £5,067,000 for silver and manufactures (£5,908,000 in 1924); and £798,000 for platinum against £1,367,000 in 1924. The average value of platinum declined from £25.32 per ounce troy in 1924 to £7.76 per ounce troy in 1930, or by about 69 per cent.



Photo] *[Yorkshire Post*

This photograph shows men controlling the flow of molten iron from the blast furnace into the pig-beds at the Hunslet yard of Walter Scott Ltd. (Leeds Steel Works), where this furnace was re-started on January 7 after an idle period of six months. The capacity of the furnace is 1,000 tons of pig iron per week. Slag, which is obtained as a by-product in the smelting operation, is ultimately utilised in the manufacture of cement. Walter Scott Ltd. are making high-class foundry and forge pig iron and also special cylinder iron.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

The Silver Situation

DURING the past few months the leading metal producers of the world have been busy studying their fields co-operatively in an effort to control prices and distribution, and by means of intensive research they have also endeavoured to find new outlets for surplus production. Silver is one of the many primary products to which the last method has already been applied with some success. The world production during the first six months of 1931 amounted to approximately 100,000,000 fine ounces, a quantity far in excess of the absorption capacity of the world's silversmiths or mints. There are no signs of a revival of bimetalism, although, under the auspices of the International Chamber of Commerce, experts from Great Britain, India, China, Japan, France and the United States met unofficially in September last to consider ways and means of stabilising the price, and the question of a silver currency is therefore not discounted. In its newer applications silver is already employed in the construction of plant for food processing, acetate rayon manufacture, mercerising and dyeing operations, and in the handling of acetic acid and caustic alkalies, but still further progress is now made possible in a study of the effect of impurities such as dissolved oxygen on the resistance of silver to attack, and a recovery in price will probably find silver firmly established as a necessity in many chemical operations.

Nickel in Electrolytic Hydrogen Plant

ONE of the outstanding features of the chemical technology of the past decade has been the development of processes involving gaseous reactions, and the production of hydrogen has become a factor of paramount importance. The filter-press type cell has many advantages as a vehicle for the manufacture of pure hydrogen, and the most widely employed electrolytic oxy-hydrogen generator in Europe to-day—the Pechkranz electrolyser—is of this type. Difficulties encountered in the operation of the earlier filter press electrolysers have been eliminated in the Pechkranz model, by means of the substitution of perforated nickel sheet diaphragms for asbestos, and the use of very efficient packing methods. The diaphragms are perforated with holes of less than 0.004 in. diameter, i.e., some 5,000-6,000 holes per sq. inch, strengthened by cross-bars attached with pure nickel rivets, and are cemented into circular iron frames. These metallic diaphragms, according to a paper by R. T. Elworthy which appears in *Chemical and Metallurgical Engineering* for December, are absolutely proof against deterioration or attack by hot alkali, and unlike asbestos diaphragms, which swell and thicken after a time, the nickel diaphragms increase in porosity in course of service. The electrodes in the Pechkranz presses are made from sheet iron, thickly nickel-plated on the anode side, a solution of 25 per cent. caustic potash in distilled water being employed as the electrolyte. The purity of the hydrogen obtained is 99.5 to 99.9 per cent., and that of the oxygen 98.5 to 99.0 per cent., without subsequent purification.

Natural Gas in Zinc Production

DETAILS of a process in which methane or natural gas is used as a reducing agent for zinc are contained in *Report of Investigations No.* 3091, issued by the United States Bureau of Mines. The process, which is expected to have an

outstanding effect on the production of metallic zinc from relatively low-grade ore, is the invention of Charles G. Maier, metallurgist at the Pacific experiment station, Berkeley, California. It follows three years of experimental study by the Bureau of Mines to determine if zinc-smelting costs could be reduced and high quality maintained. By his invention zinc of electrolytic purity is said to be producible with comparative economy. Calculations made at Berkeley enabled the prediction of the chemical combination of zinc oxide from the ore and of methane or natural gas at a certain temperature—less than 1,000°C. This is comparatively less than that required in other methods and it is vitally effective in economy of plant construction and of operation, and there are other advantages in low maintenance costs, in minimised labour, high recovery, and quality of product. The use of natural gas in the new process is expected to have many important effects. It is particularly suitable for continuous countercurrent operation, permitting high extraction of zinc without interruption. The undesirable formation of blue dust, a mixture of 10 per cent. zinc oxide and 90 per cent. of impalpable zinc powder, and difficult to control in the older dry processes, can be practically eliminated by it. There is no expense for gas-producer equipment to convert coal to gaseous fuel. The fuel economy is high, as 12,000 cu. ft. of methane at 10 cents per thousand will reduce 1 ton of metallic zinc for which 1 ton of coal would otherwise be required. The possibilities of the new method have been proved by trial at the Rare and Precious Metals Experiment Station at Reno, Nevada.

A Revival of Basic Bessemer Steel

OF the 96 papers presented in the metallurgical section of the last International Congress of Mining and Metallurgy, no less than 67 relate to the metallurgy of iron and steel. They have now been reproduced in a sumptuous volume of proceedings. The blast furnace papers, and one in the steel sub-section, by S. Surzycki, on the working of open-hearth steel charges with molten pig iron, without the addition of scrap, will possibly be of the greatest interest to English readers. Mr. Surzycki deals with the advantages of an all-in pig iron charge in the open-hearth furnace when the price of scrap is as high as that of pig iron, or higher, or when scrap is scarce. Additions up to 20 and even 30 per cent. can be made by "oreing" (the old ore process which has to so large an extent been abandoned). This, of course, further cheapens manufacture. Although in our own country scrap is still nominally cheaper than pig iron, the state of the blast furnace industry shows how imperative it is to endeavour to revert to the old methods of making steel from pig iron with its stimulation of iron ore mining and coking practice.

THE ideal process for stimulating the use of pig iron is, however, the Bessemer process rather than the open-hearth process and in view of the abundance in Great Britain of suitable ores for making basic pig iron, it is the basic Bessemer process that may be looked to for helping the British iron trade out of its difficulties. By far the greater part of the imports which so greatly harass the home trade consist of basic Bessemer steel, of which close on 17,000,000 tons were made on the Continent last year. It is the fashion to decry basic Bessemer steel, and its own advocates would not indeed claim its superiority over open-hearth steel for many purposes. Nevertheless, for some purposes it *is* superior, and tinplate makers find they make few stickers when rolling basic Bessemer tin bars down to plate, while tube makers prefer it for strip on account of its better welding properties. The Bessemer process, too, is attended with the production of a high-phosphorous slag, which is a much needed by-product. It is all to the good to learn that one of the largest iron and steel undertakings in Great Britain is seriously contemplating the installation of a large basic Bessemer steel works for the production, it is said, of close on 500,000 tons of steel yearly.

Steel Industry Co-operates with Canada

THE British steel industries' efforts to achieve co-operation with the Canadian industry were described by Mr. Julian Piggott, manager of the British Steel Export Association, who addressed the Royal Empire Society in London on January 26. In this country, he said, the British Steel Export Association, representing nearly all the leading steel makers of the country, had found itself in a particularly strong position for negotiation, whether with a foreign buyer, shipping and railway companies or competitors overseas. After visits had been paid to Canada in 1929 and 1930, which opened the eyes of the industry to the possibilities of the Canadian market, the problem presented itself how best to increase the sales of British steel at the expense of other foreign suppliers and without injuring the Canadian steel industry. The steel makers as a whole had for some time been agreed on a policy of co-operation with Dominion producers, and it remained to put this policy to the test and ascertain reactions to be anticipated from the Dominions. At the beginning of 1931 Mr. Piggott was therefore entrusted with a mission to Canada, as from experience gained on the two previous visits he had informed British makers that a policy of non-competition with the Canadian steel works was practicable. The Canadian mills had hitherto limited their production to the lighter sizes of structural sections and plates, for which a sufficient demand existed to ensure economic manufacture. The balance of the material used by the structural and engineering industries comprised those heavier lines for the manufacture of which our British mills were peculiarly adapted. It was therefore easy to aim from the start at an agreement which preserved light structural steel to the Canadian mills, while enlisting their aid in diverting to the United Kingdom as large a proportion as possible of the heavier lines Canada was forced to import.

Working Agreement on Sales Policy

HAVING convinced the Canadian buyer that British steel was every whit as good as anything in the world and that despite handicaps in transportation we could still be relied upon to deliver the goods, Mr. Piggott turned to the problem of rationalisation, and discussed with the leading Canadian producers close working agreements. This was more difficult than it would have been had the Canadian steel industry combined to the same extent as the British and there had been one central steel council. In some cases there was found to be little or no competition; in others it was possible to make some slight adjustment in our quotations so as to ensure no overlap, and a close working agreement on sales policy was evolved. In general the discussions with the Canadian steel makers took place in a most friendly atmosphere, and he hoped that when these questions came to be discussed by the experts at Ottawa, British and Canadian steel makers might go forward with an agreed policy. He thought sufficiently detailed information had been obtained for the British industry to be able to formulate certain concrete proposals which they hoped would command the support of Canadian producer and consumer alike. The tide of Imperial sentiment in Canada was running full, and we might never have such another opportunity to work out, in conjunction with Canadian industry, the problems of Imperial rationalisation which really lay at the foundation of the economic Empire.

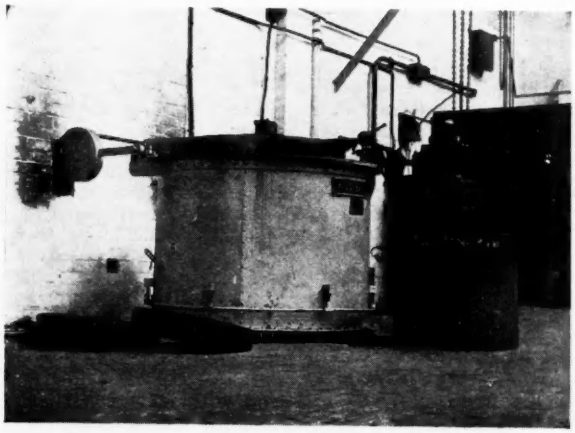
Scientific Societies' Transactions

UNLESS the publication committees of the innumerable scientific and learned societies the whole world over determine to curtail severely the scope and length of the various papers ultimately printed in their transactions there is a grave danger that those most interested in the developments described will find the task of even attempting to keep abreast of them an impossible one. This reflection is inspired by the receipt of the proceedings of two such bodies only, running into little less, in the aggregate, than 5,000 pages of closely printed matter. Many papers read before such societies are admirable in their way, and serve excellently as subjects for an evening's discussion. The accession, year by year, of new members justifies, moreover, the reading of what may be called "review papers" discussing and collating the researches on any given subject which have been made in the past, or the comparatively elementary paper or address which restates, for the benefit of the younger members or beginners, the broad principles, theory, or practice of some given subject. The society journals, issued yearly or half-yearly would be better were their contents confined to the papers, or parts of papers which really add new knowledge to a subject—the others can, and indeed should, be published in the technical papers devoted to the needs of the industries concerned. There they serve, on a wider scale, the same purpose as reading them before the Society has done, as the readers of trade and technical papers are being constantly reinforced by new subscribers to whom such papers may be really useful.

Electric Furnaces for Metallurgical Operations

Two developments of interest to the metallurgical industries are the introduction of the new Wild-Barfield electric furnace with forced air circulation and automatic charge progress recorder, by Wild-Barfield Electric Furnaces, Ltd., Elecfurn Works, Holloway, London, N.7, and the acquisition by the General Electric Co., under an agreement with Mr. Victor Stobie, of the sole rights to manufacture and sell within the Empire high frequency induction type electric furnaces in accordance with the Stobie patents.

The Wild-Barfield furnace is of the pit type and consists of a heavy metal case, brick-lagged, with heating elements evenly spaced round the interior. The heating elements are surrounded by a cylindrical heat-resisting metal sheet perforated in places. In the base of the furnace is a patent centrifugal fan, the operation of which considerably increases the rate of heating. The motor driving the fan is fixed outside the base of the furnace. To overcome uncertainty as to uniform heating, the Wild-Barfield-Foster charge progress recorder (patents pending) has been developed, and it not only controls the temperature of the furnace but automatically announces when the work being heated has reached a uniform temperature throughout the charge, and in addition a permanent record is obtained of each batch of work treated.

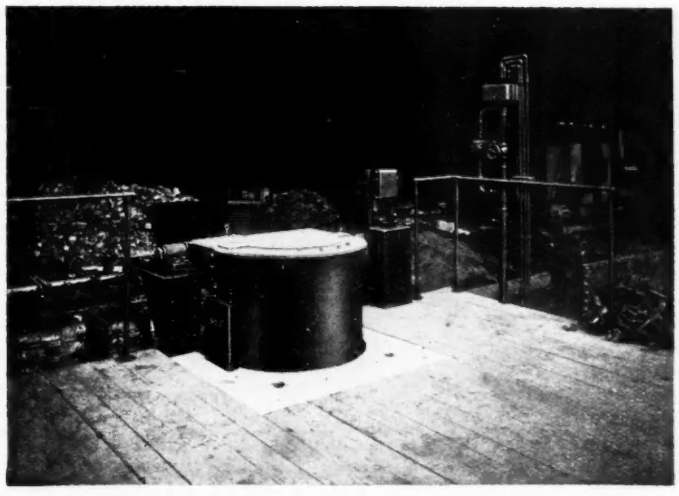


WILD-BARFIELD PIT TYPE TEMPERING FURNACE.

The Witton Furnace

The Witton high-frequency induction furnace (Stobie patents) of the General Electric Co. offers many advantages for the production of high grade steels and the melting of ferrous and some non-ferrous metals and alloys. Steels and other alloys are melted with such precision that the product is identical in composition with the arithmetical sum of the materials charged into the furnace. Temperature control is exact. Charges melted in the furnace do not take up sulphur as they do in coke or producer gas-fired crucible furnaces. High-carbon chromium and other alloys which have a strong affinity for carbon are melted without any possibility of carbon addition. The molten metal is stirred automatically, ensuring a uniform product throughout.

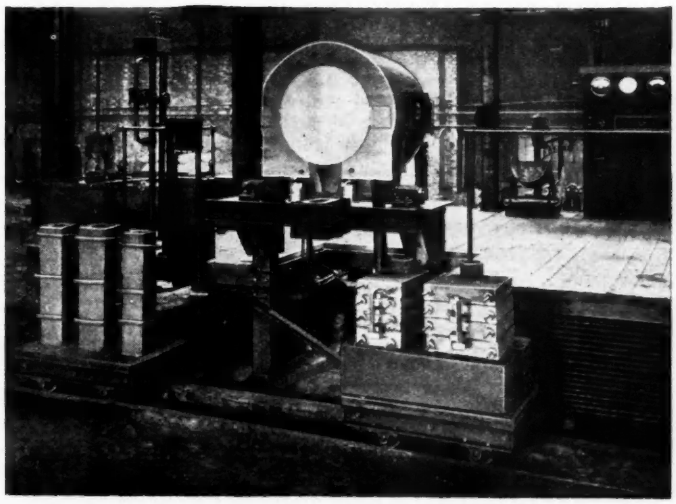
Melting costs are considerably lower than those of the older methods. Based on average rates for electric supply in industrial districts, steel, for instance, superior in quality to the best crucible steel is produced at approximately half the cost of the crucible process. The Witton furnace differs from other high-frequency furnaces in that it is not of the coreless type but is partially cored. The path for the magnetic field in a Witton furnace is mainly through thin laminations of high-silicon low-carbon steel. Instead of a magnetic field weakened by air resistance and uncontrolled as regards its distribution within the crucible, the electro-magnetic system of the Witton furnace enables so strong and centrally situated a magnetic field to be available in the crucible for inducing the current in the charge, that it has been possible for the remainder of the electrical equipment to be made entirely subservient to considerations of safety and efficiency (both electrical and metallurgical).



WITTON HIGH-FREQUENCY INDUCTION FURNACE (STOBIE PATENT). THE MELTING PROCESS.

The Institute of Metals

FOURTEEN papers covering a wide field of metallurgical work will be presented at the twenty-fourth annual meeting of the Institute of Metals in the hall of the Institution of Mechanical Engineers, Storeys Gate, London, S.W.1, on March 9 and 10. The presidential address will be delivered at the opening session on the morning of March 9, and the papers will be presented and discussed in the afternoon and at both sessions on March 10. In the evening of March 9 the annual dinner and dance of the Institute will be held at the Trocadero Restaurant, Piccadilly Circus.



WITTON FURNACE. END OF POUR.

Some Inventions of the Month

By our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Annealing

IN a heat treatment applied to magnesium and its alloys, described in Specification 357,428, International Convention date, December 11, 1929, by I.G. Farbenindustrie, of Frankfort-on-Main, Germany, the metal or alloy is immersed in a saline melt, particularly a potassium nitrate melt, with an addition of a small quantity, e.g., 1—2 per cent. of an alkali chromate, bichromate or fluoride. An anti-corrosive coating is thereby formed on the metal or alloy to protect it from attack by the melt.

METAL articles are annealed by treatment in a molten mixture of common salt, sodium or potassium carbonate, and potassium sulphate in such proportions that the ratio of sodium to potassium present is not greater than 1. An addition may be made of up to 6 per cent. of borax or sodium phosphate and up to 10 per cent. of carbonates or oxides of alkaline-earth metals or magnesium. See Specification 357,538, dated June 19, 1930, by E. Fahrländer, of Pforzheim, Germany.

Cobalt

IN a process for recovering cobalt or compounds thereof from copper pyrites, described in Specification 357,366, International Convention date, September 17, 1929, by E. A. A. Grönwall, of Stockholm, Sweden, the pyrites is roasted and melted, e.g., in a reverberatory or shaft furnace, to form a matte, the matte is Bessemerised, and the blowing is continued until most of the cobalt or cobalt sulphide has passed into the slag; the latter is then subjected to further treatment for the extraction of the cobalt or cobalt compounds. As examples of such further treatment are specified (a) lixiviation with sulphuric acid and crystallisation or precipitation of the cobalt compounds so extracted, (b) subjecting to a chloridising or sulphatising roast in admixture with a sulphur-containing material such as pyrites, with subsequent lixiviation, (c) melting with sulphides, preferably in an electric furnace, to form cobalt sulphide and recovering as a copper matte enriched with cobalt which may be again Bessemerised or roasted to form a slag to be treated like the original slag by the methods described above, (d) subjection to a reduction melting in an electric furnace to form a copper-iron-cobalt alloy which is dissolved in acid and subjected to precipitation or electrolysis.

Extracting Metals

SUBSTANTIALLY pure metals are obtained from raw materials, among which are specified bauxite, clay, leucite and sulphide ores, by a process which involves transforming them into solutions of chlorides, dissolving cane-sugar, glucose, milk-sugar, starch or other suitable water-soluble organic substance in the solutions, evaporating to dryness, and calcining the residue, preferably in a reducing atmosphere. The hydrochloric acid driven off may be recovered for use again. The organic substance is carbonised by the calcination and imparts a porosity facilitating treatment of the mixture with liquids or gases. By heating this mass to a high temperature, preferably with the introduction of reducing gases, the metal is produced. See Specification 358,000, International Convention date, June 25, 1929, by A. S. Burman, of Huddinge, and I. Rennerfelt, of Djursholm, Sweden.

Iron and Iron Alloys

A CHLORIDISING process for the purification of iron and of alloys of iron with elements forming volatile chlorides is described in Specification 358,404 (Johnson), dated March 31, 1930, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfort-on-Main, Germany. The impure metal is treated at elevated temperatures with chlorine or hydrogen chloride or gases containing them, the chlorides of the metals are separated from the impurities by volatilisation and, with or without further purification, are reduced by means of hydrogen. The chlorides may be completely or partially converted into the oxides or oxychlorides by oxidation or by treatment with steam before effecting the reduction. Examples relate to the purification of (1) cast iron, (2) ferrochromium containing about 63 per cent. of chromium, 29 per cent. of iron, and 6.8 per cent. of carbon, (3) an alloy containing 73 per cent. of iron, 7 per cent. of nickel, and 20 per cent. of chromium, and (4) ferrosilicon containing 70 per cent. of silicon. The chloridising may be effected in a rotary tube or shaft furnace and the reduction in a tube furnace.

Purifying Refractory Metals

CARBON impurities are removed from refractory rare metals, e.g., tungsten, molybdenum, niobium and tantalum by heating the metal *in vacuo* in admixture with a suitable proportion of an oxide of a volatilisable metal of which the temperature of volatilisation is below the melting point of the rare metal. The temperature must be such that the carbon will be oxidised and the volatile metal volatilised without melting the rare metal. The use of oxides of magnesium, lithium and beryllium is specified. The powdered mixture of the oxide and the rare metal may be pressed into bars and heated by passing an electric current through the charge within a sintering furnace. The carbon monoxide is pumped off and the volatile metal condenses on the cooler parts of the furnace. See Specification 358,531 (White), dated April 8, 1930, a communication from Fansteel Products Co., Inc., of Chicago, U.S.A.

The United States Lead Industry in 1931

THE lead industry in the United States in 1931 was severely affected by the depressed industrial conditions existing throughout the world since the latter part of 1929. Output of refined primary lead from domestic ores was 32 per cent. lower than in 1930, following a decline of 15 per cent. in 1930, and was the smallest recorded since 1910. Foreign primary output was 24 per cent. lower than in 1930, following a drop of 32 per cent. in 1930, and was the smallest recorded since 1921. The total refinery output of primary lead was 31 per cent. below 1930, after dropping 17 per cent. in 1930, and was the smallest since 1908. The quantity of lead indicated available for consumption in 1931 was the lowest on record since 1912, and represented a 29 per cent. decrease from 1930. The output of primary domestic desilverised lead in 1931 was about 206,000 tons; of soft lead about 143,000 tons, and of desilverised soft lead about 41,000, making a total output from domestic ores of about 390,000 tons of refined lead. Corresponding figures in 1930 were 326,801 tons of desilverised lead, 201,361 tons of soft lead, and 45,578 tons of desilverised soft lead, making a total of 573,740 tons. The output of lead smelted and refined from foreign ore and bullion was about 53,000 tons, as compared with 69,293 tons in 1930. The total primary lead smelted or refined in the United States in 1931 was thus about 443,000 tons, a decrease of about 31 per cent. as compared with the total of 643,033 tons in 1930. The output of primary antimonial lead in 1931 was about 9,000 tons, as compared with 13,711 tons in 1930.

Curtailment of Copper Production

MUFULIRA Copper Mines, Ltd., announces that, in pursuance of the agreement concluded by the principal copper producers of the world for the curtailment of production and the curtailment of production and the marketing of copper, an arrangement has been made whereby Roan Antelope Copper Mines, Ltd., and Rhokana Corporation, Ltd., will produce the company's quota of copper production for 1932. The terms of this arrangement are regarded as very satisfactory to the three companies concerned, in view of the important economies to be obtained by producing the total tonnage represented by the quotas of the three companies from two plants instead of three. In consequence of this arrangement, the starting up of the Mufulira plant will be postponed.

Industrial Fellowship at Mellon Institute

DR. EDWARD R. WEIDLEIN, Director of the Mellon Institute of Industrial Research, Pittsburg, Pa., has announced that the Lukens Steel Co., of Coatesville, Pa., has established in the Institute an Industrial Fellowship, the purpose of which is the scientific investigation of processes employed in the manufacture of steel plates. Mr. Erle G. Hill, who received his professional education at the University of California, has been appointed to the incumbency of this Fellowship. He is a specialist in iron and steel technology and was previously associate professor of metallurgy in the School of Mines at the University of Pittsburgh.

Monthly Metallurgical Section

Published in the first issue of "The Chemical Age" each month.

NOTICE.—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154 Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.*

The Future for Metallurgical Coke

The Urgent Need for Rigid Specifications

SOME observations on the future demand for coke in pig iron manufacture were given in a paper by R. A. Mott, of Sheffield University, which was read before the Institute of Fuel on the occasion of their December meeting. Steel, as a world commodity, attained in 1929 the level of output which would have been expected had the normal rate of increase in pre-war years been continued, without the disturbances of the war and post-war years. Conditions peculiar to this country prevented Great Britain from sharing in the increased demand for steel, but there is reason to believe that a change will occur which will enable Great Britain to take what may be regarded as her proper share. Should this occur, then the normal rate of development of the steel industry from 1900 to 1918 should lead to a steel production in 1940 of 16,000,000 tons. In recent years the pig-iron output has been about 80 per cent. of the steel output. This figure would probably be greater with increased steel output, but, even with the same percentage the pig-iron output would be 12,750,000 tons, and the coke requirement about 16,000,000 tons in 1940 at the present rate of fuel consumption. In other words, the coke used at blast furnaces would be twice as much as has been used on the average for the years 1928-29-30, an increase of 8,000,000 tons.

Utility of Control by Specification

The fact that the iron manufacturer has to pay more for a poorer quality of material is due to the absence of any specification as to what constitutes coke. When an improved demand for pig iron, and, therefore, for coke, arises, the iron manufacturer has no satisfactory means of preventing the coke manufacturer from controlling his coal washing and coke quenching operations less carefully, for he cannot change his source of supply, as he can in times of bad trade. It is well-known that the ash content of both coal and coke rose considerably during and after the war, but is now much reduced. There is little doubt also that, if statistics were available, the moisture contents of coke would be found to be higher during the war years than recently.

With the increases in demand for coke which are likely to occur in the future, it is desirable that the iron manufacturer should protect himself from the deterioration in coke quality which has occurred in similar circumstances in the past. The sale of coke to specification with a basic price governed by the normal laws of supply and demand, but associated with bonus and penalty clauses for quality above or below a certain standard, would ensure that the coke user would not have to pay a higher price for an inferior article. Such an arrangement would simplify the working arrangements of the coke producer and user, and would enable the iron producer to control his costs more satisfactorily.

Coke for Foundry Purposes

The market for foundry coke is a limited one and requires only about 600,000 tons per annum for inland use. The requirements are : (1) moisture should be low because of its carbon-diluting effect: (2) ash should be less than 8 per cent.; (3) sulphur should be less than 1.0 per cent. and preferably less than 0.8 per cent.; and (4) the coke should have a 1½ in. shatter index of over 90. Cokes which meet these specifications for sulphur and hardness are usually made from South Wales or Durham coals, and will meet such other requirements as the ability to produce a high combustion temperature. This factor cannot be specified, since it requires a difficult test for its assessment, but it is operative in the reputation which different foundry cokes possess.

The foundry coke users are distributed all over the country, and are generally conservative in their outlook. It is for this reason that Durham beehive coke will still bring a premium price for foundry purposes, although it has been shown that, when made from the same coal, "patent" oven coke is as effective as beehive coke for foundry purposes (*Northern Coke Research Committee Report,* 1926, 29, Newcastle-upon-Tyne). The same authority showed that coke graded 2 to 3 in. size was as satisfactory as ungraded (32 per cent. over 4 in.) coke.

In Durham, beehive foundry coke is valued 5s. to 10s. higher than "patent" oven foundry coke per ton. In South Wales, foundry coke is valued 7s. to 10s. a ton more than furnace coke, whilst "special foundry coke" is at least 5s. a ton still higher in price. There is undoubtedly a basis of quality underlying most of the reputations of good foundry cokes, for which users are prepared to pay a high price. When a cupola is giving trouble the use of certain brands of first quality foundry coke will almost certainly restore it to normal working. A sliding scale basis of payment is probably not suitable, but the competition among producers for a comparatively small trade will ensure that good quality is the basis of the price charged, although the bonuses for specially good quality are higher than for coke used for all other purposes.

Crucible Steel Industry

The beehive coke-oven industry for the manufacture of metallurgical coke apparently originated in Sheffield to make coke from small coal for the use of the crucible steel industry. Records are available which show that as early as 1805 beehive ovens were built at Sheffield for this purpose. The crucible steel industry is now only a small one, but beehive coke is still used, the coke made from the same coal in "patent" oven being less satisfactory. It has been clearly established that the preference for Yorkshire beehive coke is not founded on prejudice, but is based on the fact that such coke breaks up readily without wastage to the most suitable size for packing into a relatively small furnace, gives a higher temperature of combustion under given conditions and requires less attention for clinker removal. Yorkshire beehive coke is valued at 10s. to 15s. per ton more than "patent" oven coke for this purpose, the increased value being again based on quality.

Canadian Steel Industry

Reduced Production of Castings and Forgings

THE Dominion Bureau of Statistics at Ottawa has just issued its Report for 1930 on that section of the iron and steel industry engaged in the production of castings and forgings. During the twelve months the total output was £14,800,000, a decline of 19 per cent. as compared with the 1929 figures. Of the 340 firms in this industry, 194 were located in Ontario, 58 in Quebec, 30 in British Columbia, 30 in the Maritime Provinces and 18 in the Prairie Provinces. Their total capitalisation was £20,000,000, and their 20,499 employees earned in all £5,170,000. Among the principal products made by these firms were: heating and ventilating equipment such as stoves, furnaces, boilers, oil burners, etc., valued at £3,700,000 at the works; 200,000 tons of iron and steel pipe and fittings worth about £3,200,000; 80,000 tons of metal castings at £1,900,000; motor car parts and accessories worth £920,000; 17,000 tons of forgings at £600,000; cast iron or sanitary enamelled ware at £500,000; over 6,000 tons of bolts, nuts and rivets, and many other lines.

Electrolysis as a Competitior of Furnace Processes

Its Scope and Limitations

We give below the general outline of an article which has recently appeared in the German technical press, reviewing 50 years of progress in electrolytic practice with special reference to its status as a rival of the furnace processes for the extraction and refining of metals.

THOUGH now an indispensible agent in metal production, electrolysis has not caused the revolution nor justified the expectations raised at its inception; it still occupies a comparatively restricted place and has a severely limited scope. The exposition of the causes and the discussion of possibilities of overcoming them are particularly valuable, and are reviewed in an article by Dr. H. Wohlwihl which appears in the January issue of *Metall und Erz*. In this article the question is examined from two angles—electrolytic metal extraction from raw material, and electrolytic refining of crude metal. In the first of these fields—the extraction of the common heavy metals electrolysis has still a long way to go; only about 9 per cent. of the world's copper, and 28 per cent. of the zinc is so obtained. About 60 per cent. of the cadmium is electrolytically extracted, but the aggregate is small (approximately 1,000 tons per annum) and this is essentially a by-product from zinc manufacture. About three-quarters of the world's zinc is still marketed from furnace operations and little from electrolytic refining of crude anodes.

Basically, electro-extraction is no longer an electrochemical problem, but a metallurgical one with respect to roasting and a chemical one as regards leaching, above all, of liquor purification. Its limited scope is due to the ready fouling of the electrolytic liquors which quickly disturbs the operating conditions and contaminates the cathode products. If sufficiently pure liquors are available the problem of electrolysis is purely an economic one and the primary requirements for effective competing power are lower establishment costs for power, the consumption of which. even with high efficiencies, amounts to some 2,200-2,500 kWh. per ton of copper and 4,000-5,000 for zinc. In general, electro-extraction cannot be said to have ousted smelting methods expect under special and limited conditions. Successful zinc electro-extraction also seems to be largely a matter of special purity of electrolyte, which if not maintained may seriously disturb, if not completely stop, the operations.

Electrolytic Plant Operating Costs

Whilst basic costs for the large American electrolytic plants are given as $38-42 per ton of zinc against a normal cost for muffle processes of $45. the Tainton process shows a cost of $50 per ton. But against this it produces a high-value guaranteed 99.99 per cent. metal which commands a premium of $20 per ton, whilst by-products gain another $15. It cannot, of course, be affirmed that on these factors alone a general measure of the economic status of the processes can be based, but it indicates the variety of circumstances which may operate in affecting the rivalry between them.

Regarding the original field of electrolysis, viz., refining *e.g.*, of copper, its obvious economic and technical advantages over the older processes—furnace refining—are generally recognised and it has found more extended application. Ninety per cent. of the world's copper is electrolytically refined. Though no lead is extracted electrolytically from ore-liquors, about 12 per cent. of the total output has been refined electrolytically from crude anodes. Silver in overwhelming proportion (83 per cent. of the U.S.A. production) and also the greater part of the bismuth output (practically 100 per cent. in the U.S.A.) were refined electrolytically. Of the total nickel output—some 64,000 tons per annum—at least half is produced by electrolysis following furnace processes.

Purity of Product

The superiority of electro-refining is embodied first in the requirements of electrical industry (which absorbs about 70 per cent. of the total copper output), demanding the very highest conductivity that only metal of the greatest purity can satisfy. and secondly, in the returns from gold and silver values originally in the ore and ultimately concentrated in the metal. These two objects are achieved simultaneously with a completeness and perfection unattainable by other means. This purity of final product, however, necessitates a comparatively high grade of raw material to work on, constituting one of the disabilities of electrolysis as a rival of other processes, since this can only be attained when a furnace operation has taken over the greater part of the burden and pre-refined the anode-copper to 98-99 per cent.

In this respect indeed, electrolysis does not seem to have advanced technically, on recalling the expectations of the earlier workers that means would be found for electrolysing anything and everything. High-quality copper had been obtained from the crudest black copper, and the then marvellous power of the electric current with the possibilities of selective solution and separation of metal from a complex mixture misled to the idea that means were at hand for resolving into their constituents the most difficult combinations such as occur in ores, mattes and speisses. by means of a single electrolytic operation. This was never realised. Nevertheless there is recalled how, during the war, when such attempts were based on stern realities and supported by economic conditions not available at normal times, much work was done at the German metal factories whn supplies became scarce, in attempts at electrolytic recovery of various ingredients of already used alloys. Electro-deposition from bronzes and copper nickel alloys was conducted on a large scale, metals carrying 17-20 per cent. tin and contaminated with lead and antimony were electrolysed direct or else pre-refined by bessemerising and then cast into anode plates.

Furnace Methods for Pre-refining Stages

Technically, the operations were ultimately more or less successful; as an economic proposition for normal times quite impossible, and it was established that though electrolysis might play its part for the final stages. it was usually more economical to conduct the cruder pre-refining operations by furnace methods up to as late a stage as practicable. A full account is given of the treatment of the often highly arsenical nickel bearing copper-solutions produced at these refineries. Eventually electrolysis was resorted to and reasonably uncontaminated copper ultimately produced, but accumulation of nickel and arsenic in the liquors threatened the wreck of the processes until the Schopper process brought success. This method involves the use of sulphur dioxide under pressure; the arsenic is completely reduced to metal and separates perfectly from the strongly acid mother-liquors in a very pure form (99 per cent.), which renders comparatively simple the later separation of the nickel.

With respect to gold, technically, the electro-refining process approaches perfection, yet sulphuric acid parting of silver-bearing gold is still general, because this operation is more convenient for treating the usual small lots as and when required whilst the values are quickly obtained in a form that can be seen, handled and marketed. Electro-refining of such bullion is more suited to central stations working on a steady and continuous basis, when its superiority is unquestionable as regards cleanliness, completeness, and especially in conjunction with platinum group metals.

The Betts' Electrolytic Process

Of electro-refining as applied to lead, two propositions are laid down: first, that the early expectations attached to the Betts' process using fluorsilicic acid electrolyte, of its ultimately superseding the older fire-refining processes, have not been fulfilled, nor seem likely to; and secondly, that up to the present there is no process that can compete with it for the separation of bismuth from lead, though the possibilities of the new Kroll process using metallic calcium and still in the development stage are not to be disregarded. That the Betts' process yields a satisfactory product is evidenced by its satisfying the requirements as a raw material for the white-lead industry which are particularly drastic. The Harris process (a furnace method utilising the oxidising power of nitrate additions to the slags) as applied to the treatment of work-lead carrying 1 or 2 per cent. of impurities such as tin, arsenic or antimony, constitutes an exceptionally satisfactory method, as each constituent is removed with completeness and is separately recovered in a marketable form.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

Commercial Production of Gallium

THE *Chemiker-Zeitung* for January 2 announces that the Vereinigte Chemische Fabriken, at Leopoldschall, Stassfurt, manufacturers of the rare element rhenium, have succeeded, after lengthy experimental trials, in isolating gallium by a method which enables them to produce the metal at one-twentieth of its former cost, which a year ago stood at about 175 gold marks per gram. A way to the commercial development of this remarkable metal is therefore opened. Considerable attention has already been given to the properties of gallium, chiefly on account of the long thermal range of stability of its liquid phase; the pure metal actually melts at about 30° C. and boils at a temperature above 2,000° C. It has therefore been used for filling quartz thermometers for registering temperatures above 500° C. Some of these have already been sent to the Physikalisch-Technische Reichsanstalt for standardisation. Gallium can also be used to replace mercury in vapour arc-lamps for use in analytical work. Indeed, its application in this direction should lead to important results, on account of the wide range in frequency in the lines of its spectrum, which extend from the ultra-violet to well into the red region. It has also been used in the form of alloys with other metals to replace the dental amalgams, which are said to have produced some harmful results, and for the production of special optical mirrors.

Increasing Applications for Cobalt

COBALT-tungsten-chromium alloys have come into wider use in recent years as they are among the most abrasive alloys known. It has been found that, by means of the electric arc or acetylene torch, they can be deposited on certain surfaces to protect them from wear, and also to build up the worn surfaces of cast iron or steel. The use of cobalt in permanent steel magnets is also increasing, as such magnets are much smaller and lighter than ordinary steel magnets of equal power. Cobalt-tungsten-carbide alloys are being most successfully employed in machining metals at high speeds, and in increasing the life of cutting tools. An alloy of this type, manufactured in the United States, may be said to consist of particles of tungsten-carbide which have been cemented together by metallic cobalt using an electric furnace of the resistance type. The durability of this material is much greater than that of high-speed tools, and it has been used in cutting threads upon glass, in cutting concrete, porcelain and nickel steel. For drilling in concrete or rock it is less expensive than the diamond drill, and is stated to cut a smoother hole than the hammer drills. A similar alloy is manufactured in Germany. The hardness of these cobalt-tungsten-carbide alloys in the Brinell scale varies between 1,250 and 1,400, compared with 1,000 for the hardest steel.

Leaded-Bronze Bearing Metals

A STUDY has been made at the United States Bureau of Standards upon the effect of casting temperatures and of additions of iron on the properties of bronze consisting of 80 per cent. copper, 10 per cent. tin, and 10 per cent. lead. In addition to determinations of hardness and microstructure of this leaded bronze, several test methods were employed which simulated certain phase sof actual service conditions. These tests included deformation under pounding, and resistance to wear when dry and in the presence of a lubricant. As bearings are usually above room temperature while in operation, a considerable number of the above tests were conducted at elevated temperatures. In bronze founding it is seldom possible to pour all castings at the same temperature because the metal is drawn from the furnace at various temperatures and cools in the ladle during pouring operations. A study of the effect of different casting temperatures therefore aids the manufacturer in producing better castings and at the same time helps the consumer by predicting what variations in properties may be expected of bronzes cast at various temperatures. In the study of the 80: 10: 10 bronze referred to above, when cast at temperatures varying from 1,850° to 2,120° F., it was found that as the casting temperature, the grain size, and the resistance to wear were increased, the hardness decreased slightly, and a slight tendency was noted for more even lead distribution. Casting the bronze at about 2,000° F., caused a decrease in the resistance to impact or lowered the toughness as shown by the notch test. The deformation under pounding was a minimum in specimens cast between 1,900° to 2,000° F. Additions of iron up to 0.5 per cent. decreased the resistance to wear and increased the hardness. When the iron content exceeded 0.3 per cent., segregation of the lead particles occurred, the grain size was reduced, and a decrease was noted in the notch toughness.

Volume Changes During Casting

THE American Foundrymen's Association and the United States Bureau of Standards have been co-operating in a study of the shrinkage of metals during casting and the influence of such shrinkage on the production of sound castings. This work is reported in the January issue of the *Bureau of Standards Journal of Research*. The total shrinkage of the metal or alloy for casting purposes has a profound influence on the foundry practice for that particular metal. It affects the size of the pattern required to give the casting of definite dimensions; the design of the casting: the size, type, and location of risers and feeders needed: the liability of castings to crack in the mould, and the soundness of a pressure test. The present paper describes a method which has been developed for measuring the volume changes undergone by metal or alloy with change in temperature as it cools from the liquid state to ordinary temperatures. It defines three types of shrinkage to be considered: (1) Shrinkage of the metal in the liquid state; (2) shrinkage during solidification; and (3) shrinkage in the solid state. The specific volume-temperature curve for liquid metal is constructed from data secured by application of the crucible immersion method, that is, by filling a crucible of known volume with the liquid metal at known temperature. Since the mass of the sample of liquid metal is the same as that of the sample at room temperature, the liquid specific volume may be readily calculated. Data on the contraction of solid metal were obtained by direct observations of the change in length with change in temperature of a sand-cast bar. The difference between the specific volume of the liquid metal and of the solid metal at the melting point is regarded as the volume shrinkage undergone during solidification. In the case of an alloy, shrinkage during solidification occurs over a range of temperatures.

The Revival of Bessemer Steel

THE observations of Sir William Clare Lees, president of the Association of British Chambers of Commerce, at the annual dinner of the National Federation of Iron and Steel Manufacturers on February 19, regarding the re-establishment of basic Bessemer steel plants in this country, has aroused interest in the iron and steel trade. The Bessemer process was quite generally employed in this country, where it was originally evolved, until some time before the war, when it was almost completely dropped because the steel produced was not of such consistent quality as steel manufactured by the open-hearth process. The British Engineering Standards Association have for a long time insisted on a specification with which Bessemer steel did not comply and which is admittedy much higher than those in use abroad. Sir Clare Lees' argument, that if British steel manufacturers are to secure a much larger volume of export business they must offer consumers abroad a steel of not quite such high quality as they normally make if the consumer wants it, was originally advanced at the meeting of the Iron and Steel Institute last year when Mr. Benjamin Talbot urged the production of Bessemer steel in this country. Basic Bessemer steel can be used very successfully by the re-rolling trades, and Continental imports of Bessemer steel have been largely used by the tinplate industry and for manufacturing light sections. The open hearth steel process not only produces a higher quality steel of greater consistency than is achieved by the Bessemer process, but is a more expensive process. Much of the reputation which British steel has achieved abroad is due to the industry's determination to produce the highest possible quality, but for many purposes steel of a lower quality is quite satisfactory, and the suggestion that Bessemer steel plant should be erected in this

country would indicate that the steel manufacturers have decided to compete with foreign makers for this class of business.

Corrosion Resistance and Protective Coatings

WHILE for a large number of purposes non-ferrous metals and alloys giving resistance to corrosion are needed in various branches of engineering, iron and steel are the cheapest of all metals employed, and the most prone of all to suffer from corrosion. Hence of the vast amount of research work which has been done during the last ten to twenty years on corrosion, the bulk has been in relation to iron and steel, and methods of rust-proofing, or the provision of non-rusting ferrous materials have had more attention devoted to them than to any other corrosion preventative methods. There emerge, from the researches already carried out—apart from the use of paints, lacquers, etc.—three main methods of preventing iron and steel from rusting. These are the spray or cementing processes of coating iron and steel with a protective metal, electro-deposition, and the use of alloys which, of themselves, resist corrosion and therefore need no further protective coating. It is not surprising, therefore, to find that competition is extraordinarily keen between these three modes of rust prevention.

Possibilities of Zinc Coatings

CHROMIUM plating has, at present, a serious rival in zinc, which although not usually regarded as a decorative metal, can be used as a coating for iron and steel that only an expert can distinguish from chromium plating itself. Zinc may be applied to iron and steel by spraying, or the iron and steel may be electroplated with zinc. Both these processes are cold-zincing processes, and in the cold no iron-zinc alloy is formed between the top zinc coating and the base metal. This is claimed, on the one hand, to be an advantage, as the iron-zinc alloys are somewhat brittle, and, on the other hand, to be a disadvantage, as the pure zinc coating is less adherent. The causes of embrittlement of iron and steel when zinc coated are obscure and appear due rather to the preliminary coating of the base rather than to the results of actual plating. The spraying of zinc is hardly a commercial proposition for the zincing of sheet and wire, for which the great bulk of zinc coating is required. There are two alternative methods of hot zincing, the hot-dipping process known as galvanising, and cementation. Hot zincing is invariably attended with the formation of an inter zinc-iron alloy, although Bablik has recently described a modification in which the formation of this intermediate layer is avoided. The alternative hot-zincing process is cementation, better known as sherardising. Electro-deposited zinc coatings can be controlled to give very uniform and exact thicknesses of zinc provided the plating is not too thick, otherwise it peels or cracks. Hot dipping is difficult to control, and galvanised coatings are much thicker, often, than they need to be. Sherardised coatings can be controlled very accurately, and a recent development of this process is its application to sheets which it has not, hitherto, been possible to "sherardise" in the barrels or drums employed for less bulky articles. A new process of wet sherardising is also being developed. Sherardised articles always have the zinc-iron alloy layer, and it has yet to be shown that this is really brittle, or embrittling. On the other hand, it is intensely adhesive, and as zinc is anodic to iron, and the zinc alloys are likewise anodic to iron, but cathodic to zinc, the coatings are doubly protective in the best sense of the electro-chemical theory. Moreover, the richer zinc zinc-iron alloys have a brilliant lustre, take a fine polish, and are untarnishable by ordinary atmospheric agencies.

The Need for Curtailment of Transactions

REFERENCE was made in last month's Metallurgical Section to the need for a severe curtailment of the scope and length of the papers printed in the transactions of the innumerable scientific and learned societies throughout the world if the danger that those most interested in the developments described find the task of even attempting to keep abreast of them an impossible one to be averted. We urged that the societies' journals might be confined to papers or abstracts which really add new knowledge to a subject, and that the other papers might be published in the technical papers devoted to the needs of the industries concerned. It devolves, then, on the publication committees of the societies themselves to keep down the bulk of their transactions by limiting the papers printed, or reprinted for permanent record to those recording new facts, or presenting old facts in some new light. Judicious abstracting and systematic cross indexing will do the rest, with some indication of whether the matter referred to is general, historical, or actually a record of something new. We would suggest that all tabular matter presented by the authors should be most carefully scrutinised so that the results or inferences may transpire readily, and the reader can be relieved of the task of scanning pages of figures which embody confused or discordant results derived from incomparable experiments and observations. This particular reflexion is the result of wading through columns of figures in a recent paper giving the results of tests on the relative corrosion resistance of a number of materials at different field stations. The criticism raised is, unfortunately, applicable to many publications of this nature. Industrial concerns such as the Mond Nickel Co., or the British Aluminium Co., which have well found and ably-directed information bureaux, or similar organisations for collecting and collating the literature on a given subject, may be able to give the necessary time, and go to the necessary expense of winnowing the wheat from the chaff, but for the ordinary scientific worker the task is rapidly becoming a hopeless one.

Removal of Antimony from Lead

THE removal of antimony in the processes of smelting and refining lead is being studied by the United States Bureau of Mines at their Pacific Experiment Station, Berkeley, in co-operation with the University of California. Several papers covering the physical constants of antimony oxides have already been published. The direct oxidation of antimony in lead bullion by the tetravalent or pentavalent antimony oxide in the semi-liquid oxide skin is also being investigated. It appears that the trivalent oxide in the slag is oxidised by the atmosphere of the softening furnace to a higher valent form, which in turn migrates to the surface where it oxidises the antimony dissolved in the molten lead. Due to valency changes the antimony in the oxide skin may, therefore, act as an oxygen carrier from the atmosphere to the molten metal.

Beryllium in Industry

A Survey of its Present-day Uses and Prospects

DURING recent years considerable attention and research has been directed towards bringing some of those substances which are generally described as "rare metals" into the sphere of commercial use. Particularly has this been the case with those which are light in weight, and which therefore suggest themselves, either alone or as alloys, for employment both in the construction of aircraft and in the automobile industry. Amongst these metals, beryllium stands out prominently, for not only is it much lighter than aluminium and almost as light as magnesium, but its melting point is much higher than that of either of these light metals. In order to provide authoritative information about this new metal and its ores and to act as a stimulus to its commercial use, a monograph surveying the present state of knowledge concerning beryllium has been produced by the Mineral Resources Department of the Imperial Institute in their well-known series of monographs on economic minerals. This book (*The Mineral Industry of the British Empire and Foreign Countries—Beryllium and Beryl;* H.M. Stationery Office, pp. 26, price 6d. net) contains an outline of the processes for extracting the metal, a summary of its properties and of its principal alloys, a statement on production and prices, and an account of the distribution of deposits of beryl —the only beryllium ore of which adequate supplies are known to exist. It concludes with an up-to-date bibliography of the principal literature on the subject.

The Imperial Institute publication indicates that the British Empire is in a strong position to hold its own in respect of this new metal, if only adequate financial resources can be found to follow up the work of the Empire's investigators and mineral explorers, and to make use of the very valuable properties which beryllium has been found to impart to some of the other commercial metals. At present the metal is being produced commercially only in Germany and the United States.

Tin in Industry

Its Outlook Reviewed in the United States

THE value of tin used in the United States is much greater than that of any other mineral product which is furnished almost entirely by importation. Tin imports, according to a report recently issued by the United States Bureau of Mines, have annually exceeded £12,000,000 in value, but domestic mine production was worth less than £2,000 for the last year recorded. Moreover, the net imports of tin exceed, in value, those of any other mineral product and the metal is among the ten leading commodities imported. The principal tin-consuming industries are food-packing, motor car manufacture, and building. The packing of food is relatively stable from year to year but the motor car and building industries are subject to wide variation in tin consumption.

The Tin Producers' Association continued its efforts to control tin production and stabilise its price until the inability of the Association to obtain adequate co-operation from tin mines, led to a movement to have the governments of the principal tin-producing countries enforce a definite curtailment programme. After several months of negotiations, the governments of the Federated Malay States, Bolivia, the Netherland East Indies, and Nigeria agreed to accept quotas for the total mine production of their respective countries for a period of three years after March 1, 1931, and to enforce these quotas on their tin producers by law. Since then, Siam has been induced to join the quota plan. The principal consumers of tin, with the exception of the British, have not been represented in the formulation of the quota plan. The smelting of imported tin concentrates was an important domestic industry in the United States from 1916 to 1924. Since then the inability to obtain clean concentrates to mix with the refractory Bolivian concentrates ended the United States tin-smelting industry, at least temporarily. The enormous local market, however, coupled with the proximity of Bolivia, should allow the United States to build up a large tin-smelting industry if a process for handling Bolivian concentrates economicaly, without admixture of clean ore, could be devised.

The Search for Tin Substitutes

A few years back, when the price of tin was approximately double what it has averaged recently, the opinion was widely expressed that the reserves of tin, marketable at the prices then current, were very limited. This resulted in intensive research for substitutes. The field for making important substitutions seemd very promising because at that time tin was the highest priced metal in the common base-metal group. Since the fall in the price of tin, the search for substitutes has diminished. Domestic consumers depend on substitutes as one of their most effective means for maintaining reasonable tin prices as well as one of their principal means for continued production in the event that war should curtail the flow of tin to the United States. Considerable study has been given to the problem of finding a substitute for tin-plate in the canning industry, but to the present time no tin-free container has been developed that can offer serious competition to the tin can. Aluminium is being used as a substitute for tin in the manufacture of foils and collapsible tubes. Because of its low specific gravity a given weight of aluminum will make much more foil of a given thickness than an equal weight of tin but this advantage is offset in some tin foils by rolling from bars containing a lead core. Tin foil is also meeting severe competition as a food and cigar wrapper in "Cellophane," a transparent cellulose product, and in various waxed papers.

Chrome Vanadium Steel

Its Use in the Construction of Pressure Vessels

ADVANCES in chemical engineering practice during the past two decades have been in large measure devoted to the application of higher temperatures and pressures. Many of the processes, however, either require only moderately high temperatures or do not exceed such temperatures because of the impossibility of securing, at present, sufficiently large units of sound highly alloyed steels, so often necessary to withstand the high-temperature corrosive attack. The low-alloy steels have consequently served for many processes operating at moderate temperatures under mildly corroding conditions, although at very high pressures. They have, of course, also replaced carbon steels in vessels operating at room temperature, principally to effect weight saving. Among these low-alloy steels, chrome vanadium has been so thoroughly successful as to warrant a description of some of the major applications in the January issue of *Vancoram Review*, which is published by the Vanadium Corporation of America.

Steels containing from about 0.10 to 0.45 per cent. of carbon with one per cent. or somewhat more of chromium and approximately 0.20 per cent. vanadium have been preferred not only for their excellent mechanical properties at both room and moderately elevated temperatures but also because of their uniformity, even in large masses, and their adaptability in all fabricating operations. These desirable qualities account for the production or large ingots for conversion not only into forgings but also into both light and heavy plate for the construction of welded units. The soundness and hot-workability of these very large ingots and the exceptional mechanical properties of electrically-welded equipment of large size have also been especially noteworthy.

Several forged steel vessels are illustrated in the article referred to, one of these vessels having been produced from a steel ingot with the following limits of composition : Carbon 0.3 to 0.4 per cent., chromium 1.25 to 1.50 per cent., vanadium 0.18 per cent. (minimum), phosphorus 0.045 per cent. (maximum) and sulphur 0.045 per cent. (maximum), the finished product having a yield point of 40,000 lb. per sq. in. (minimum), and a tensile strength of 80,000 lb. per sq. in. (minimum). This particular vessel was of cylindrical shape 42 in. bore, 55 in. outside diameter and 42 ft. 6 in. long. After testing at 6,000 lb. per sq. in. it was placed in service in the petroleum industry at a working pressure of 3,500 lb. per sq. in. and a maximum outside shell temperature of 750° F., where it has rendered most satisfactory service for a period of several years.

Canada's Mineral Production in 1931

A General Decline Reported

ACCORDING to the preliminary estimate of the Dominion Bureau of Statistics, Canada's mineral production in 1931 was valued at £45,500,000, as compared with £56,000,000 in 1930. In spite of the decline in prices of most minerals, however, the mining industry of Canada is in a robust condition and is in a position to take full advantage of the expected upward economic turn.

Gold production, stimulated by lower commodity prices and the present exchange situation, increased by 27.5 per cent. over 1930 and assisted materially in offsetting the loss occasioned by the drop in the prices of other metals. Production in the metals group was valued at £23,300,000 as compared with £28,500,000 in 1930. The output of gold, which was the greatest yet recorded in the history of Canada, rose from 2,102,000 ounces valued at £8,700,000 in 1930 to 2,680,000 ounces valued at £11,100,000. The last figure does not include the premium received by the gold mining companies due to the present premium on the rate of exchange. As in other years production in 1931 was valued at the standard rate of 2,067 cents per ounce.

Copper production amounted to 290,581,000 lbs. valued at £4,700,000, as compared with 303,478,000 lbs. valued at £7,600,000, a decline of 37 per cent. in value and 4 per cent. in quantity over 1930.

The production of lead ammounted to 267,851,000 lb. valued at £1,400,000, as compared with 332,894,000 lbs. valued at £2,600,000. The production of nickel amounted to 66,104,000 lb., which is considerably under the total for 1930; silver amounted to 200,404,000 ounces valued at £1,200,000, as compared with 26,444,000 ounces valued at £2,000,000; zinc amounted to 236,861,000 lb., which is 11 per cent. under 1930 production.

Iron and Steel Production in Canada

THE Dominion Bureau of Statistics at Ottawa reports that, during the year 1931, a total of 420,038 tons of pig iron was produced, as compared with an output of 747,178 tons in 1930, and 1,080,160 tons in the record year 1929. The past year's output included 311,850 tons of basic iron, 80,892 tons of foundry iron and 27,296 tons of malleable iron. Blast furnace charges for this production included 735,878 long tons of imported iron ore, 221,106 short tons of limestone and 437,013 short tons of coke.

Some Inventions of the Month

By our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Alloys

ALLOYS for permanent magnets, described in Specification 356,169, International Convention date, March 8, 1929, by Deutsche Edelstahlwerke Akt.-Ges., of Krefeld, Germany, comprise nickel-free steels containing 0.75—1.15 per cent. of carbon, 2.02—4.9 per cent. of chromium and either 0.5—1.5 per cent. of molybdenum or 0.5—2 per cent. of tungsten or 0.6—2 per cent. of molybdenum and tungsten together. Cobalt up to 1 per cent., vanadium up to 0.5 per cent., manganese up to 0.25—0.9 per cent., silicon up to 0.05—0.35 per cent., and the usual small amounts of phosphorus and sulphur may also be present. The specification as open to inspection under Section 91 (3) (a) of the Acts covers a considerably wider range of alloys.

Alloys : Electroplating

TARNISH-resisting alloys of silver and iridium with the silver predominating are described in Specification 359,014 (Triggs), dated July 11, 1930, a communication from Oneida Community, Ltd., Oneida, New York, U.S.A. They are obtained either (1) by melting the metals together, (2) by depositing the iridium electrolytically on the silver and thereafter diffusing it into the silver by heat, (3) by depositing the two metals simultaneously on a suitable base, and stripping from the base, or (4) by depositing alternative layers of iridium and silver on a base, diffusing the iridium into the silver by heat, and stripping from the base. Other metals, such as palladium or zinc, may be added.

Cementation

BATHS for use in the cementation of articles of iron, steel or alloys thereof, especially alloys containing nickel, chromium, vanadium, molybdenum or tungsten, are described in Specification 359,483 and addition thereto, 359,486, International Convention dates, July 22, 1929, and February 1, 1930, respectively, by Deutsche Gold-und-Silber-Scheideanstalt vorm. Roessler, of Frankfort-on-Main, Germany. According to Specification 359,483, the bath contains barium cyanide together with not less than 40 per cent. and preferably above 50 per cent. of barium chloride at 850-950° C. and may be prepared by adding a suitable proportion of alkali cyanide to barium chloride or to mixtures containing 40 per cent. or more of barium chloride. Substances which lower the melting point of the bath, such as sodium or potassium chloride, and a small proportion of carbon may be added. Access of air may be prevented by a surface layer of graphite or charcoal. According to Specification 359,486, strontium salts, such as the chloride, bromide or carbonate, replace the barium chloride wholly or in part.

Hardening Steel

IN a process for the surface hardening of steel or steel alloys an oscillatory electric field is produced between a metallic piece and the steel, etc., the dielectric being an atmosphere adapted to yield carbon or nitrogen or both. The steel, etc., is maintained at a temperature below the lowest of the critical points during the treatment. Alloys which may be hardened are those containing one or more of the elements nickel, chromium, aluminium, boron, manganese, molybdenum, silicon, titanium and tungsten. See Specification 360,833, International Convention date August, 9, 1929, by Soc. d'Exploitation des Procédés Mahoux, of Paris, assignees of A. P. Mahoux, of La Garenne, Seine, France.

Refining Metals and Alloys

THE use of iodine for refining metals and alloys is described in Specification 357,539-40, dated June 19, 1930, by May & Baker, Ltd., and G. S. Higginson, of London.

357,539. Refining material in the form of one or more iodides, e.g., of potassium and/or barium, is placed in or applied to a vessel containing the molten metal, and is thereupon decomposed by the intense heat, the free iodine so produced being caused to pass through the metal. About 1—2 parts of iodide are used to 1,000 parts of molten metal.

357,540. A cartridge or casing containing the refining material is inserted into the molten metal. The cartridge may be of cardboard, iron, aluminium, synthetic resin, etc., and may be attached to the end of a rod which is inserted in the metal.

IN a process for producing ingots or castings free from gases and from oxides by subjecting the molten metal to a reduced pressure, the metal is subjected to a preliminary overheating, preferably with stirring, to a temperature at least 50° C. higher than the casting temperature to cause it to absorb large quantities of gas. The reduced pressure may be applied intermittently. If the temperature of overheating is very high, some cold metal, already treated by the process, may be added, before or during the application of the reduced pressure, to cool the mass more quickly to the casting temperature. The treatment of steel, copper, nickel, magnesium and alloys thereof is specified, e.g., nickel, casting at 1,480° C., is heated to 1,530° C. or higher, and an alloy containing 95 per cent. of magnesium, 3 per cent. of aluminium, and 2 per cent. of manganese, 3 per cent. of aluminium, 750° C. See Specification 358,563, dated July 10, 1930, by A. Godefroid, of Berlin.

THE removal of bismuth from metals, especially lead, containing it as an impurity is effected by the addition of calcium and magnesium or calcium and barium to form a dross containing a substantial part of the bismuth, according to Specification 360,789, International Convention date March 29, 1930, by American Smelting and Refining Co., of New York, assignees of J. O. Betterton, of Omaha, Nebraska, U.S.A. The addition may be in the form of a calcium-magnesium-lead or calcium-barium-lead alloy. The dross containing bismuth is removed and the bismuth content of the lead is further reduced by selective freezing. The calcium, barium, etc., are finally removed as chlorides by treating the lead with chlorine under a zinc chloride slag.

Coating with Metals

ARTICLES of iron or steel are bronzed by immersion in a hot aqueous bath containing caustic soda, litharge, potassium cyanide, ferrous sulphate, lead sulphate and manganese dioxide for 5—15 minutes at a temperature not above 135° C. See Specification 361,095, dated November 6, 1930, by V. Rosso, of Liège, Belgium.

Rustproofing Ferrous Metals

IRON, steel and alloys thereof are provided with a rust proof layer by heating for up to 24 hours at 300—900° C. and preferably at 400—600° C. in a non-oxidising gas containing phosphorus. Suitable gases are (1) hydrogen phosphide with or without an admixture of nitrogen, hydrogen or carbon monoxide, (2) any non-oxidising gas containing phosphorus vapour. Treatments of wrought iron and of chromium aluminium steel are described. See Specification 359,987 (Johnson), dated May 2, 1930, a communication from I. G. Farbenindustrie Akt.-Ges., Frankfort-on-Main, Germany.

The United States Zinc Industry in 1931

UNFAVOURABLE industrial conditions throughout the world continued to affect the zinc industry in the United States in 1931, and the year witnessed the lowest production on record since 1921. The output of primary metallic zinc from domestic ores was 40 per cent. lower than in 1930, following a decline of 20 per cent. in output in 1930. Apparent deliveries of primary zinc to consumers were 21 per cent. below those of 1930 which, in turn, were 28 per cent. below apparent deliveries in 1929. As evidence that zinc producers were attempting to meet existing conditions in their industry, smelter stocks were reduced from 167,293 tons at the end of 1930 to 135,600 tons at the end of 1931, a drop of 19 per cent. This reduction is noteworthy for it was achieved in spite of a large decrease in consumption. The decrease in production of primary metallic zinc for 1931 as compared with 1930 (498,045 tons) amounted to 41 per cent. In addition to the output of primary zinc, about 21,000 tons of redistilled secondary zinc was produced, as compared with 34,849 tons in 1930. Of the total output of primary material in 1931, 82,000 tons was electrolytic zinc. A total number of about 85,000 retorts was reported at the 17 zinc smelters that operated during all or a part of the year. Of that number, 19,000 retorts, less than one-fourth, were reported in operation at the end of November.

Monthly Metallurgical Section

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NOTICE.—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, *Bouverie House, 154 Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.*

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

The Tin Restriction Scheme

THE two leading non-ferrous metals—copper and tin—are now facing adverse circumstances due to heavy stocks in hand, uneconomic prices and the absence of an anticipated increase in consumption. Producers of both metals are, therefore, making strenuous endeavours to restrict output still further. In the case of tin a fairly comprehensive restriction scheme, which has been in operation for over twelve months, under Government control, has so far failed to bring about the desired improvement, notwithstanding the fact that the output quota under the original scheme was reduced further as from the beginning of the present year. The International Tin Committee is therefore discussing the possibility of a third cut in production. The amount suggested is 8,420 tons per annum, equal to one month's output. This, coming on top of the 15,000 tons cut in January, is likely to cause fresh resentment among producers, many of whom are already compelled to work their mines on an attenuated basis. There is little doubt, however, that the proposed new cut will be put into operation at the beginning of May or June by the latest, although a final decision will not be taken until the International Committee's next meeting in London, on April 22. Total world stocks of tin at the end of February were 54,266 tons, as compared with 44,080 tons at the end of February, 1931. This increase has taken place in spite of a cut in production from 172,000 tons in 1930 to 144,000 tons in 1931. Against this fall of 28,000 tons in production, there was an estimated drop of about 40,000 tons in consumption, and this latter unsatisfactory feature has continued during the early months of 1932. Consumption in January and February of the present year was only 16,402 tons, as compared with 19,917 tons for the same period last year.

The Copper Situation

THE copper restriction scheme, unlike that of the tin producers, is an entirely voluntary one, and it remains to be seen how long the new and cheap producers who could conceivably work at a profit at present prices will be willing to help shoulder the burdens of the older undertakings. In face of increasing stocks and falling prices, copper producers have been persuaded to agree to a further curtailment of output from 26½ per cent. to 20 per cent. of capacity. It is hoped that this new rate of production will reduce the world's monthly output from 85,000 tons to between 65,000 and 70,000 tons. The market, however, has not shown much enthusiasm for the new scheme, and the export price of copper in America is now down to 6¼ cents per lb. One of the biggest firms of producers announces the shutting down of two of its most important mines, as there is little hope of immediate improvement because of the existence of heavy stocks. At the end of 1931 it was estimated that about 800,000 tons of copper remained in the hands of producers, plus a substantial tonnage in the hands of consumers. The normal holding of surplus stocks is considered to be about 350,000 tons, and even if the agreed new cut in production is faithfully carried out it will require to be combined with a material improvement in consumption to bring about an adjustment of stocks to normal proportions. A revival of world industrial activity would stimulate a demand for copper as for other commodities, but it is doubtful whether mines having a big potential future output in prospect will consent to restriction for an indefinite period.

A New Rustproofing Process

A PROCESS has been developed by the I. G. Farbenindustrie utilising phosphoric acid as a rust remover and for rustproofing. The iron to be treated is first freed from corrosion products by immersion in a 15 per cent. phosphoric acid bath, and, after washing with water, it is then immersed in a 1 to 2 per cent. phosphoric acid bath which is kept as hot as possible. The etched iron removed from this bath dries by the heat acquired whilst in the phosphoric acid bath and a very thin rustproof phosphate layer is formed. For the simultaneous removal of grease, other chemicals are added to the phosphoric acid. The I. G. surface protecting process—"atramentverfahren"—consists of a preliminary preparation of the iron surface, production of the "atrament" layer and an after-treatment. The iron from which rust and grease have been removed by known methods of treatment, are transferred to the "atrament" bath which contains 3.5 per cent. of a concentrated metal phosphate solution known as "atramentol." The temperature of this bath is maintained at 98° to 100° C. and the iron is allowed to remain immersed for about one hour, following which it is rinsed in water and allowed to dry.

New Aluminium Corrosion-Resisting Alloys

ALTHOUGH aluminium of a degree of purity equal to 99.5 per cent. or higher resists corrosion much better than does the crude metal, its chemical stability is still inadequate for many uses and its relatively low mechanical strength limits its applications. Hitherto it was generally considered that introduction of alloying metals necessarily lowers the corrosion resistance of pure aluminium, but recent developments have proved that this is erroneous, as several binary alloys have been found to equal very pure aluminium in its resistance to attack. Among the least familiar improvements in this respect is the use of small quantities of manganese in very pure aluminium, permitting the usual tolerances with respect to quantity of such impurities as iron and silicon in the aluminium. One new binary aluminium manganese alloy, containing less than 2 per cent. of manganese, is claimed to be considerably harder than 99 per cent. aluminium and has a much higher tensile strength. Its resistance to corrosion is also much greater than that of pure aluminium, so that its service life is longer. This alloy is being successfully used for making kitchen-ware, fermentation vats and articles subject to sea-water exposure. It is anticipated that it will also find extensive application for plating some of the light metal alloys.

Developing a Wider Use for Magnesium

THE Aluminium Company of America and the I. G. Farbenindustrie of Germany have formed a company to be known as the Magnesium Development Co., with the object of promoting the development and utilisation of magnesium and its alloys. It is planned to license suitably equipped fabricators on a low royalty basis, in return for which licensees will receive all necessary detailed technical assistance. Prior to 1914 no magnesium was produced in the United States, but war-time prices and demand led to a number of companies to undertake production. Following the return of normal business, however, only the Aluminium Company of America and the Dow Chemical Co. remained in the field. About five years ago the Aluminium Company discontinued production and centred its activities on developing the fabrication and sale of the metal. The Dow Company also placed emphasis upon the production of magnesium, but continued work on fabrication as well. In Germany magnesium has been produced and fabricated for a much longer time than in the United States, and a considerable amount of experience and patent literature

has accumulated. At times there has been great similarity in the lines of work undertaken by the American and the German concerns, with the result that there was a constant danger of infringement of patents. In order to obtain the most rapid development of this difficult field, it therefore appeared to be a desirable step for these two organisations to cast their lot together and to combine experience and patents.

Nickel Cast Iron for Paper Mill Equipment

AT the February meeting of the Technical Association of the Technical Association of the Pulp and Paper Industry, J. S. Vanick and F. L. LaQue discussed the actual and potential applications of nickel alloy cast iron in various parts of paper mill equipment. Their paper, which has been published in *The Paper Trade Journal*, January 28, is arranged to show the application of the modified materials throughout the sequence of operations, from raw to finished product. The course of the research work reported has been dictated by the knowledge that corrosion, heat and wear are the factors most potent in causing deterioration of cast iron in the paper mill, while properties such as strength, stiffness, thermal conductivity closeness of grain, density and the ability to take a fine polish also influence the operation of mill equipment. The results show that high-nickel cast iron may be relied upon to effect very substantial increase in the life of equipment used in the acid and sulphate of soda processes, and in parts working in contact with washed pulp, white water and bleach liquors, and the recommendations made are supported by detailed experimental data. The advantages of corrosion-resisting cast irons for the chemical sections of paper-making plant have their counterpart in the improvements effected in constructional cast irons (used for drier rolls, heads, calender rolls, gears, sprockets, etc.) by the addition of minor amounts of nickel and chromium.

Weld Decay in Nickel-Chromium Steels

A RECENT contribution to the literature of "weld decay" in nickel-chromium austenitic steels (*Amer. Inst. Min. Met. Eng. Technical Publication* No. 464) deals with the question from the point of view of association between magnetic susceptibility and resistance to inter-granular corrosion. The work reported covers the effect of heating for 10 mins. at temperatures within the range of 1,000°—1,600° F. (540°—870° C.). This test was considered to be a suitable criterion, since it was assumed that in most welding operations the time of heating would be considerably shorter and that materials which would satisfactorily withstand this test could be relied upon for welded structures to be exposed to active electrolytes. A large number of modifications of "18/8" chromium-nickel steels was made, and the effect of additions of tungsten, molybdenum, silicon, titanium and vanadium was studied. A solution of 10 per cent. H_2SO_4 and 10 per cent. $CuSO_4$, used boiling, was employed as the testing medium. Effects of quenching and reheating temperature are discussed and a correlation is made of the amounts of ferrite in the annealed steels and their relative resistance to intergranular attack. The prevailing theory of carbide precipitation is discussed and the results of the present study are reviewed in support of the theory that the cause of intergranular corrosion is incipient carbon separation from solid solution, rather than carbide precipitation.

The Testing of Castings

THE discussion on "the testing of castings" to which concluding session of the annual meeting of the Institute of Metals was devoted, on the afternoon of March 10, has contributed little to the solution of this complicated problem. The opening address by Dr. Rosenhain left, to the ensuing discussion itself, the drawing of any definite conclusions from the considerations of the numerous factors involved. The discussion, however, wandered somewhat, although Dr. Rosenhain had been careful to distinguish between the requirements of reception, or what he termed "specification testing" and "control testing"—the methods employed by manufacturers to enable them to ascertain and to maintain the requisite quality of their castings. Here, of course, resides the ultimate ambiguity. Manufacturers can control and maintain, by established tests, the quality of the metal they put into their castings, and while that ensures to a fairly well known extent the soundness of the molten metal to be poured, it does not and cannot ensure the ultimate soundness or strength of the castings themselves. Even the methods of testing the metal, used to elicit certain properties which it may be desirable that it, *quae* metal, should possess, may fail entirely to reveal how the castings themselves will behave, or even the soundness or strength of the metal as distinct from its behaviour as a separate "cast" test piece.

Cast-on versus Separately Cast Test Pieces

OPINIONS are divided as to the relative advantages of the separately cast test piece, and of a test piece "cast on" to the casting itself, and subsequently separated for the purpose of the test. Actually neither will serve, definitely, to indicate the nature of the casting itself; both are, in effect, quite different castings, poured in circumstances that are far from identical with those in which a given casting has been poured. Only a geometrically identical casting could give results which can afford real indication of how the casting may be expected to behave, and only then if the tests applied are similar to the service conditions expected of the casting itself. Not even the testing to destruction of one or more castings in a given batch can really serve to establish the strength or soundness of the rest; the most it can do is to establish a presumption. Reliance can be had only on careful mixing, melting and pouring; the provision of adequate heads and risers; the elimination of entrapped air or gas forming constituents in the mould; and a properly designed mould. If the discussion before the Institute of Metals showed anything it was that there is no omnibus method for testing castings; some properties, such as porosity, can be tested by specific methods, but the aggregate of properties cannot be tested and must be taken on the general repufation of the foundry.

American Metallurgical Awards

THE American Institute of Mining and Metallurgical Engineers, at their meeting held in New York, February 17, formally awarded the James Douglas Gold Medal for achievement in non-ferrous metallurgy to C. H. Mathewson, of Yale University, for "his scientific contributions to the art of working and annealing non-ferrous metals." C. T. Eddy, of the Michigan College of Mining and Technology, received the Alfred Noble Prize of $500 for a "paper of particular merit contributed by a member of any of the four founder engineering societies before the author is 30 years old." Professor Eddy, who is 28 years old, received this award for his paper on "Arsenic Elimination in the Refining of Native Copper." The J. E. Johnson, Jr., award for meritorious research in iron and steel was given to Ora E. Clark, of Hamilton, Ohio, who has "developed an exceptional insight into the reactions of an iron blast furnace."

International Tin Proposals

THE International Tin Committee met at the Bolivian Legation, Paris, on Tuesday, March 22, when Sir John Campbell presided. The committee considered a proposal that a further cut in production of 8,420 tons a year (corresponding to one month's production) should be made with effect from May 1 or June 1. A final decision on this point will be taken at the next meeting. The committee have been informed by the Committee of Control of the International Tin Pool that they considered it unnecessary during the past month to increase their holding, which stands at 21,000 tons of metallic tin.

Proposed International Silver Conference

BANKERS, economists and government officials of eight nations are being asked to express their views on the question of holding an international silver conference. According to information from Washington, the sub-committee of the House of Representatives, which has been studying the silver problem, has now despatched a document on the subject to Sir Robert Horne; Mr. Montagu Norman, Governor of the Bank of England; Sir Charles Addis, director of the Bank of England; Sir Herbert Holt, President of the Royal Bank of Canada, and Sir Osborne A. Smith, managing governor of the Imperial Bank of India.

Fuel Oil in the Metallurgical Industry

Its Advantages in Heating Processes

ONE of the most important subjects among the processes involved in industry to-day, is that of fuel economy in heating operations. Since the war the great strides made by our metallurgists in the science of materials have made it essential for the heating operations carried out either in the manufacture or preparation of these metals and alloys to be placed on a scientific basis. In the old coal furnace, which depended on weather conditions, the skill of the fireman and variable fuel, it was impossible to control temperature and atmosphere to the close degree necessary for modern production methods. It was then realised that furnaces, which previously had been given little thought, were just as important as, if not more important than, the other units in the chain of production. New fuels were required which could be harnessed to the work to be done, and controlled within exact limits, and to-day these fuels are being used successfully to an increasing extent for furnace firing of all descriptions.

In considering the best fuel to use for any heating operation, various factors besides the basic one of cost per heat unit must be taken into account. There is the efficiency with which the heat can be applied to the metal; the convenience factor, which includes accuracy of control and ease of handling, certainty of supplies, ease of storage and delivery and elimination of waste. A properly laid out fuel oil storage and distribution system in a works will give invariable results at the furnace and, in one valuable respect, is claimed to be superior to town's gas which, unless properly governed, is subject to noticeable pressure variations due to the fluctuating demand from the mains. Apart from its cheapness and convenience in application fuel oil is in some cases the only accurately controlled fuel available. In view, therefore, of the wide use which is being made of liquid fuel for nearly every metallurgical process, a brief description of some of its applications will no doubt be of interest to our readers.

Non-ferrous Metal Melting

For the melting of bronzes, brass and aluminium, oil-fired crucible type or reverberatory open flame type furnaces are widely used. The reverberatory pattern is usually made in sizes of from 160 lb. to 600 lb. (brass capacity), and the melting costs on bronze are about 3d. per 100 lb. melted. The crucible type furnace is made in sizes of 60 lb. up to one ton (brass capacity), and is employed in cases where it is not considered advisable to allow the products of combustion to come into contact with the molten metal. Oil-fired crucible furnaces can be operated very economically, fuel consumption varying according to the size of charge and the metal handled, but being lower than can be obtained with other fuels. Metal loss is also the lowest possible and operating charges are reduced to a minimum. Oil has been used in this type of furnace for the melting of pure nickel, for which a temperature of 1,600° C. is required; the reverberatory type of furnace would not be suitable owing to the effect of the products of combustion on this metal.

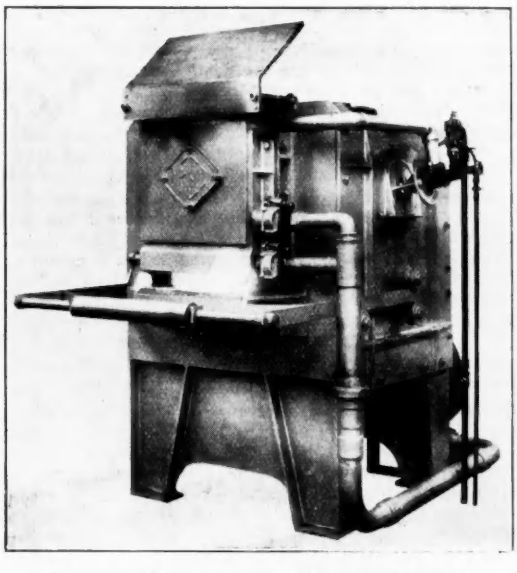
The outputs obtained with oil-fired reverberatory furnaces are quite remarkable. One 160 lb. capacity furnace occupying a floor space of a little over one square yard, will do the work of five pit fires, turning out a charge of gun metal every 25 mins., throughout the working hours, to produce castings of first class quality. Another similar furnace of 300 lb. capacity, on actual test in a large engineering works, starting from dead cold in the morning melted in 9 hours 50 mins., 16 charges of metal totalling 5,120 lb. in weight; the total oil consumption on this duty being 44 gallons.

Iron and Steel Melting

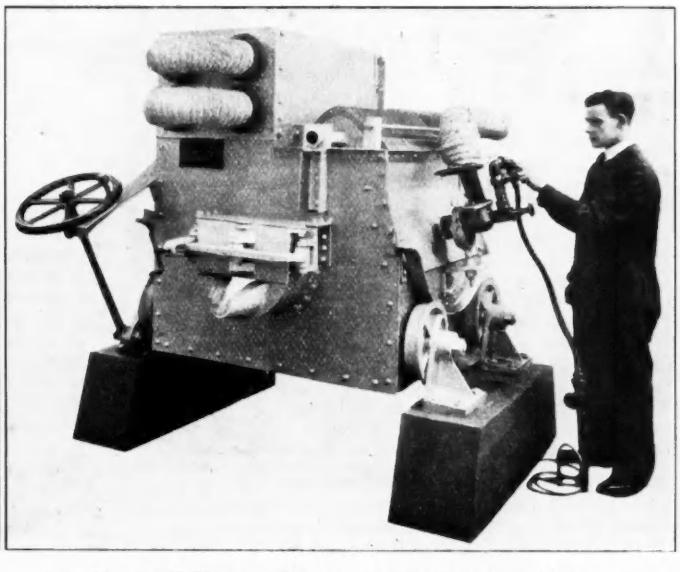
With the rapid development of special irons and alloy steels, oil-fired furnaces up to 30 cwts. capacity are proving extremely useful. They are easy to operate, quick in action and it is possible to obtain with them the desired carbon content in the resulting product. It will be appreciated that the iron in an oil-fired furnace is not in intimate contact with the carbon in the fuel as it would be in a coke fired cupola and for this reason it is possible with oil fuel to obtain specified results without difficulty. An oscillating or semi-rotary furnace has proved very suitable for melting ferrous metals and, in order to obtain economy at the high temperatures required, the furnaces are of the recuperative pattern, using highly preheated air for combustion.

Heat Treatment Furnaces

Possibly, however, the various oil-fired heat treatment furnaces which have been developed recently are of the greatest interest. For heat treatment of steel and also brass parts, the new "International" radiant wall type furnace gives remarkable results. This furnace can be operated with a reducing, neutral or oxidising atmosphere, over a temperature range from 550° C. to 1,000° C. The atmosphere in the



OIL-FIRED RECUPERATIVE FORGING FURNACE. (INTERNATIONAL FURNACE EQUIPMENT CO., LTD.)



"MIDGET" MELTING FURNACE FOR CAST IRON. (BRITISH REVERBERATORY FURNACES, LTD.)

furnace chamber is quite homogeneous, and delicate articles, such as brass strip and black sheet, can be annealed at the appropriate temperature without any scale whatever. These operations can be carried out in practice with a 14 per cent. CO_2 content in the products of combustion with consequent high fuel economy.

Of larger heat treatment furnaces, mention may be made of muffle and semi-muffle types for annealing of copper tubes, copper wire, brass strip, etc. These furnaces are fired with a number of oil burners on either side and are of the continuous type, with outputs from one up to 3 tons per hour. Cleanliness, both of the shop floor and of the atmosphere, is a valuable feature where sensitive metals, such as copper and brass, are handled, and the oil furnace compares favourably with producer gas in this respect. Fuel oil costs about 1s. 6d. per ton of metal annealed at 550°/650° C.

Forging and Welding

The use of fuel oil is now standard practice for all important reheating processes where steel parts of small size are handled. The very extensive stamping and forging trades, and the manufacture of bolts, nuts, spikes, rivets, etc., are carried out almost entirely with oil. No other fuel will give the rapid outputs required in modern works. The oil furnace is always on top of its job; it never keeps anyone waiting, the heat of the metal is always the same. The bolt and nut trade of the Midlands was probably the first to adopt the use of liquid fuel, some works having been using it for over 30 years. Many firms also use oil for plate heating, whilst the manufacture of all kinds of garden, edged and other tools is carried out to a great extent with oil-fired furnaces. The tube trades are likewise extensive users of oil. All the electrical conduit made in this country is welded in oil-fired furnaces, whilst oil is also used for tube annealing, socket welding, flanging, expanding, etc. One conduit welding furnace running at 1,450° C. gives an output of 1,100 $\frac{3}{8}$ in. conduit tubes per shift on an oil consumption of 14 gallons per hour, costing approximately 3s. 6d., whilst one medium-sized oil furnace for socket welding took the place of a whole shop full of old fashioned coke fires.

Vitreous Enamelling

One of the processes which has grown rapidly of late years is the vitreous enamelling of various steel or cast iron articles, such as parts of weighing machines, gas cooking stoves, petrol pumps, signs, baths, etc. These articles are fired in muffles at a temperature of from 700° C. to 900° C. to fuse the enamel, and oil is used almost exclusively. Cleanliness is a most important feature in a shop where this work is carried out, as the slightest trace of dust settling on the articles, after they have been coated with damp enamels before firing, causes objectionable marks, particularly with white articles. Here the use of oil ensures absolute cleanliness and freedom from dust and dirt due to smoke or ashes, and perfect control of temperature in the muffles, by automatic means if desired. The normal oil consumption on a standard muffle for this class of work, or size about 12 ft. × 4 ft. 6 in. × 2 ft. 6 in. would be about 6 gallons per hour, costing 1s. 6d.

Oil has also displaced other methods almost entirely, for the smelting of the enamel before grinding and making into paste for application to the articles. The smelting is carried out in a rotary furnace at a temperature of 1,500° C., and the high radiative power of a short oil flame, together fith the absence of ash, renders it ideal for this purpose. Many enamel works now use this method, which gives a better quality product, particularly in the case of white enamel, and almost twice the output of a coal furnace of the same size. One of these rotary smelters of 700 lb. capacity melts 4 charges totalling 2,800 lb. in 9 hours, whereas the old coal furnace took 13 hours to melt one ton of enamel. The oil fuel cost is approximately 8d. per 100 lb. enamel (melting time only), or 9¼d. per 100 lb. including getting the furnace up to temperature before starting to melt.

Japanning Processes

Under the heading of stoving and japanning come a variety of drying processes accomplished at low temperatures up to 500° F., which are carried out in continuous ovens, with the aid of an oil-fired air heater. This method is enormously superior to the old intermittent ovens fired with gas; extensive savings in time of drying together with greatly reduced fuel cost having been obtained. Nearly all the motor manufacturers use these air heaters for drying off mud-wings and a host of other articles, while the same process is used for pram parts, wheels of all kinds, etc. There is likely to be a large extension of the use of oil-fired air heaters for this class of work. Another process under this heading is the printing and lacquering of tinplate. This work requires delicate handling, particularly where white enamel is concerned, and oil firing is being widely adopted where economy, combined with perfect results, is required.

Galvanising Baths

The problem of heating galvanising baths is one which has given much trouble and many attempts have been made to devise a system which will give even heating and, at the same time high output from the bath. All the evils attending normal galvanising bath operations are traceable to uneven heating, which causes high dross loss and low bath life, as well as inequalities in the thickness of spelter on the articles treated. It has been found from experiments that a galvanising bath should be heated through the sides and that more heat should be transferred through the top half of the sides than through the bottom portion. A recent application of oil fuel, embodying novel features of heat distribution, marks a great advance in the heating of these baths.

Minerals Separation, Ltd.

Prospects of Flotation and Segregation Processes

THE twenty-seventh annual ordinary general meeting of Minerals Separation, Ltd., was held in London on March 23, when Mr. Francis L. Gibbs (chairman of the Company) presided. The profit for the period under review amounted to £33,948, as against £54,658 in the previous year, and a final dividend of 10 per cent., equivalent to 2s. per share, was declared. This, with the interim dividend of 5 per cent., which was paid in October, 1931, made a total distribution of 15 per cent., equivalent to 3s. per share for the year.

Dealing with the company's operations during the year, the chairman said that although the company's income from royalties had shown a decrease, bearing in mind the general depression which had overshadowed the metal industry throughout the year, the returns could not be considered unsatisfactory. The copper segregation plant which was erected at the Alaska Mine of Southern Rhodesia Base Metals Corporation, Ltd., was completed in February, 1931, and after running experimentally to establish conditions, the operations from the beginning of April until towards the end of June were continuous; 3,484 tons of oxide ore were treated, of an average grade of 5 per cent. copper, yielding 222 tons of concentrates with an average grade of 68.2 per cent. copper, which represents a recovery of 86.91 per cent. copper. Towards the end of the period the grade of concentrates was as high as 85.8 per cent. copper.

Research and experimental work in connection with flotation and segregation processes has been continued throughout the year at the Company's London experimental works, and several improvements have been made in the processes, mainly in the direction of extending their scope in the treatment of more difficult ores. In this connection it has been demonstrated that gold and silver present in oxide copper ores are readily recovered with the copper when the ores are treated by the segregation process. Large-scale tests have also been carried out on refractory gold ores by a combination of modern standard methods and flotation.

World Copper Agreement

THE agreement reached at Brussels on March 22 between the copper producers is now understood to be definite and comprises practically all the big producers of the world. This agreement provides for production on the basis of a 20 per cent. capacity operating rate, as compared with the present 26½ per cent. rate. The new rate is expected to reduce the world's monthly output of copper from 85,000 tons to between 65,000 and 70,000 tons, and will probably bring production below the present rate of consumption, thus reducing surplus stocks.

Utilisation of Scrap Brass

The Recovery of Copper as Copper Sulphate

SINCE it was first introduced for use in dyeing and calico printing the manufacture of copper sulphate has been improved by modern systems of production. Latterly these have principally devolved upon methods of reclaiming copper sulphate from waste metal which is secured in the form of scrap brass, turnings, etc. This brass may or may not contain lead and tin, but is usually rich in copper, of which it may contain 60 to 70 per cent.

In practice these wastes are first made into a " matte " by a preliminary process, which consists in mixing the waste with a proportion of saltcake and coal dross and subjecting it in crucibles to a red heat. The proportion of salt cake it employed depends on the copper content of the brasses, but a copper matte containing 20 per cent. sulphur is kept in view. The amount of coal dross used is approximately one third of the saltcake. The wastes, saltcake and coal dross are usually ground up to an intimate mixture in a pan mill. The crucibles used may be either clay or graphite and the largest possible size is employed, since the mass on heating is guilty of frothing whilst expelling gases. Where there is a larger demand for copper sulphate, a small reverberatory furnace is employed as by virtue of the open shape of the bed, no harm or loss is sustained by the swelling of the mass.

Recovery of Tin

The older process, or rather the process most frequently used for the conversion of brass waste to copper sulphate consists in roasting the metal to oxidise the copper, and then dissolving directly in sulphuric acid. In such accumulations of scrap, tin is present which would thus be completely lost and therefore any means of reclaiming the tin is worthy of consideration. Pyrites could be substituted for saltcake, but this would not convert the tin to a soluble form. Sodium sulphide compounds alone are able to effect this, but sodium sulphide is more expensive than the crude saltcake and coal dross.

After fusing the foregoing mixture it is tapped into iron moulds and ground up without delay so that little or no oxidation of fused sulphide to sulphate is allowed to take place. The mass is ground through a 1/16 in sieve and placed in an iron tank and boiled with water. The tin which exists as sodium sulpho-stannate is quite soluble in the excess of sodium sulphide. The liquor after settling is run through an open cloth filter, whilst the " matte " itself is passed through a vacuum filter. This mass on drying in the air crumbles to powder, which saves the cost of further grinding for the wet copper sulphate plant, and it is simply rubbed through a 1/30 in sieve. The tin is then recovered from the liquors by re-precipitation with waste acid left over from the wet plant. The fusion converts the copper to a condition of almost pure copper sulphide. Zinc does not form a proper " matte," a small proportion of it joins the copper whilst the remainder is burnt off. Lead which is in the brass is converted to sulphide by the surplus salt cake, but a deficiency of the latter can cause it to be precipitated from the mass as metal.

Dissolving and Crystallising

Having prepared the " matte " in the foregoing manner its solution in sulphuric acid is simple and direct. Iron pots heated by a coal fire in a small brick frame are used. Concentrated sulphuric acid is run into the pots and raised almost to boiling point, when the matte is charged. The mass is stirred by copper bars and dissolves comparatively rapidly. The mixture is poured whilst still hot directly into a lead-lined vat containing hot water, covered on top by a lead roof. Direct contact of the hot acid liquor with the hot water gives rapid solution to the mass. The boiling is continued for a short time and the liquor allowed to cool so that lead sulphate may settle out, from which the liquor may be syphoned off without the need for filtration, and is transferred to an evaporating tank. Here a lead-lined wooden vat is used with lead pipe coils on the bottom through which steam passes at about 70 lb. per sq. inch pressure. and copper strips are placed in the tank to neutralise the surplus acid.

Evaporation is continued until the specific gravity reaches 1.4. The blue liquor is then run into lead tanks, in which strips of lead are hung, and allowed to crystallise. Slow crystallisation favours the formation of large crystals, whilst any sediment carried is given full opportunity to separate out. A period of three days is allowed for tanks of 300 gallons to crystallise. The mother liquor is then run into a large well and if sufficiently pure may be re-used. The crystals as chipped from the sides of the tanks and the lead strips are placed in a centrifugal, washed and " whizzed," giving a product of 98 per cent. purity.

Adhesion of Vitreous Enamel to Metal

Researches in the United States

FOR more than a year a ceramic investigation of the Engineering Experiment Station, Ohio State University, has been in progress, having as its object a fundamental study of the mechanics of adhesion of enamels to metals. For the most part this study has been confined to sheet steel and cast iron, but copper and the precious metals had not be neglected, according to a brief report by Professor R. M. King (Ceramic Engineering Department, Ohio State University), which appears in the February issue of our American contemporary *The Ceramic Age*.

The history of the art of enamelling ferrous metals does not indicate that any distinction was made between the type of enamel which was placed in direct contact with the metal and that superimposed upon the contact enamel. To-day, in the enamelling of ferrous metals, cast iron and sheet steel, we call the contact enamel a " ground coat," " grip coat," sometimes a " slush coat," and the enamels subsequently applied "cover coats." At the present time general practice is to use ground coats for sheet steel and dry process cast iron, and single coats for wet process cast iron. The ground coats used for dry process cast iron are nothing more than transparent lead-borax glasses. The sheet steel ground coats differ from sheet steel cover coats in two pronounced respects, namely, (1) they contain one or more special " metallic oxides " which promote adhesion of the enamel, and (2) they do not contain large amounts, if any at all, of materials such as the fluorides, antimony oxide, and zinc oxide which promote opacity in cover enamels and decrease adherence.

In ground coat enamels, the "metallic oxides" used usually are cobalt oxide and nickel oxide. Manganese oxide is sometimes used but has little value in promoting adhesion. The most satisfactory oxide for this purpose is cobalt oxide. Nickel oxide can be substituted for part of the cobalt oxide but when used without cobalt oxide it makes the ground coat very unreliable. When partially substituted for cobalt oxide three or four times as much nickel oxide as cobalt oxide must be added to obtain the same adhering power. It has not been found necessary to add these metallic oxides to cast-iron enamel to obtain satisfactory adherence. This difference is usually attributed to the porous nature or rough surface of cast iron but may be merely due to the fact that cast-iron pieces for enamelling are usually much thicker than sheet steel pieces and hence the enamel is not subjected to the many strains and stresses that cause failure in sheet steel enamels.

In any investigation of this nature the fundamental method of procedure is to study the effect of all attendant variables upon the property or properties which are the chief concern of the problem. These variables are the enamel composition, including commercial ground coat, compounds promoting adhesion, compounds decreasing adhesion, and amount of the compounds; the metal base, ferrous and non-ferrous; and the atmosphere in which enamel is fired, whether air, oxygen, nitrogen, hydrogen, or other gases. This investigation, however, has as its ultimate aim the improvement of ground coat enamels through the increasing of adhesion and the elimination of defects.

Some Inventions of the Month

By our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Alloy Steels

IN making air-quenching steel of the type containing one or more elements such as nickel, chromium, molybdenum or tungsten, with or without titanium, vanadium, boron or copper, a thermo-aluminic process is used in which the additions intended to remain in the steel are incorporated in the thermo-aluminic mixture, while the deoxidising agents, such as ferro-manganese or ferrosilicon are added only during the progress of the reaction. The alloying elements may be added partly as shavings or grains of a steel alloy and partly as richer material, *e.g.*, nickel, ferro-nickel or iron alloys containing up to 70 per cent. of chromium, molybdenum or tungsten, or oxides of nickel, chromium, molybdenum or tungsten. Carbon may be supplied as shaving or grains of cast iron containing 2-4 per cent. of carbon. See Specification No. 361,390, C. L. Lelachaux, Gennevilliers, Seine, France.

Chromium Plating

THE resistance of iron or steel articles to rusting is increased by applying a phosphate coating containing a metal, *e.g.*, copper, having a different electric potential from iron, and subjecting the coating to the action of a solution, preferably hot, containing the chromic acid radicle, *e.g.*, a solution of potassium chromate of bichromate or chromic acid. See Specification No. 362,746 (Pyrene Co., Ltd.), a communication from Parker Rust Proof Co., Detroit, Michigan.

A PROCESS for producing a protective coating of chromium upon a foundation metal is described in Specification No. 363,954 of British Thomson-Houston Co., Ltd., London, assignees of G. H. Howe, New York. The foundation metal is heated in the presence of, but out of contact with, metallic chromium. For this purpose the metals are placed in a porous vessel which is packed in an inert medium containing a small amount, *e.g.*, about 2 per cent., of a substance, such as ammonium chloride, which dissociates on heating with formation of an acid capable of combining with the chromium. In an example an iron article is placed in an alundum tube together with, but out of contact with, some chromium metal. The tube is plugged at each end with asbestos paper and placed in an iron container, the space between being packed with an inert material, such as alumina, containing a small amount of ammonium chloride. The container is then heated in a furnace to about 1,000° C. for about 2 hours. Some chromium may be mixed with the alumina.

Concentrating Ores

IN a froth flotation process for concentrating ores there is added to the pulp, in conjunction with the usual frothing directly connected by two sulphur atoms. Specified substances are ethyl disulphide, isoamyl disulphide, *n*-amyl-disulphide, phenyl disulphide and cresyl disulphide and specified frothing agents are (a) cresol, (b) cresol and calcium oxide, (c) pine oil, (d) pine oil, sulphuric acid or sodium carbonate, and paraffin gas distillate. See Specification No. 362,366, Minerals Separation, Ltd., London, assignees of W. Trotter, San Francisco, and E. W. Wilkinson, Berkeley, California.

Extracting Metals

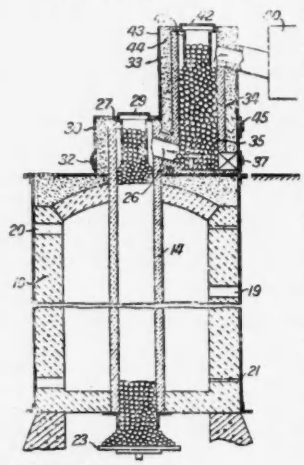
SOLUTIONS obtained in cyanide extraction processes are treated, while maintaining them alkaline, with zinc sulphate. A zinc cyanide precipitate and a cyanide-free solution are thus obtained. Gold and part of any silver present may then be recovered from the cyanide-free solution by precipitation on metallic zinc, the solution being preferably first acidified. The original cyanide solution may be treated in the usual manner for the recovery of metal values prior to the treatment with zinc sulphate. See Specification No. 362,861, H. T. Durant, Crawley, Sussex, and H. L. Sulman and H. F. K. Picard, London.

Magnesium

MAGNESIUM is obtained by reduction of magnesia with a metal, *e.g.*, tungsten, of melting point above 1,600° C. according to Specification No. 361,957, of Hirsch Kupfer und Messingwerke A.G., Messingwerke, Germany. The magnesia is heated with the tungsten and with sufficient carbon to produce an atmosphere of carbon monoxide, and the magnesium volatilises and is condensed. The tungsten oxide formed may be reduced, *e.g.*, by hydrogen, and the resulting tungsten used to reduce a further quantity of magnesia.

Extracting Zinc

FURNACES for the extraction of zinc, provided with means for eliminating lead and other undesired metals from the zinc vapour, are described in Specification Nos. 361,605-6, of the New Jersey Zinc Co., New York, assignees of E. H. Bunce and G. T. Mahler, Palmerton, Pennsylvania. According to Specification No. 361,605 the lead eliminator chamber 33 communicates at one end with the externally heated reduction chamber or retort 14 and at the other end with a chamber 40 in which the purified zinc vapour is further treated, *e.g.*, is condensed or converted into oxide. The eliminator is an upright conduit with imperforate walls, and a column of lead-eliminating material, *e.g.*, graphite, charcoal, coal, coke, silicon carbide, clay refractories, etc., passes continuously through it; it is preferably provided with means for regulating the temperature. Alternatively the zinc vapour may pass downwards through the eliminator. In a modification the eliminator is a vertical prolongation of the retort and the agglomerated charge of ore and reducing agent constitutes the eliminating materials. According to Specification No. 361,606 the eliminator is disposed directly above the retort, but is supported independently thereof, so that the expansion and contraction of the retort produce no objectionable strains in the structure of the eliminator.



361,605

Magnesium and its Alloys

ARTICLES made of magnesium or of alloys rich in magnesium are protected against corrosion by a chemical treatment described in Specification 353,415 of H. Sutton and L. F. de Brocq, of Farnborough, Hants. The cleaned articles are immersed in a solution containing (a) one or more chromates or bichromates of alkali or alkaline earth metals, ammonium or substituted ammonium, and (b) one or more salts of such metals or ammonium bases. The solutions used must be free from manganese compounds, aluminium compounds, and salts of the heavy metals. Guanidine sulphate is specified as a salt of a substituted ammonium base. The bath may be acid, neutral, or alkaline, and when an acid bath is used the articles are preferably subjected to an after-treatment comprising immersion for a few minutes in a hot or cold solution of alkali metal chromates and bichromates.

NON-FERROUS metals and alloys are refined by introducing into the molten metal or alloy the vapour of a volatile halide of vanadium, e.g., vanadium tetrachloride. The process is especially applicable for refining aluminium and its alloys. See Specification 352,671, D. R. Tullis, Bearsden, Dumbartonshire, and P. Oakley, Liverpool.

Precious Metal Alloys

ALLOYS containing from 60 to 100 per cent. of gold or silver and up to 30 per cent. of a metal of the platinum group, with or without other precious or non-precious metals such as copper, are converted into alloys capable of substantial age-hardening by the addition of about 0.02-5 per cent. of one or more of the metals iron, nickel, cobalt or manganese, or about 1.8 per cent. of one or more of the metals zinc, cadmium or chromium, or metals from both of these groups. Age-hardening is effected by heating to about 900° C., quenching, and annealing at about 550° C. for varying periods. See Specification No. 361,893, Deutsche Gold-und Silber-Scheideanstalt vorm. Roessler, Frankfort-on-Main.

Metallurgical Section

May 7, 1932.

Current Topics

Metal Refining in Canada

THE Dominion of Canada is now one of the world's largest exporters of refined metals. In a paper contributed by Mr. R. W. Brigstocke, of Toronto, at the recent annual meeting of the Canadian Institute of Mining and Metallurgy, it was pointed out that a few years ago copper, zinc, lead, nickel, silver and other metals were exported from Canada mostly in the raw state. To-day, all these metals are refined in Canadian plants and a surplus of pure metals is exported, thus retaining in Canada the fullest possible percentage of their ultimate value. In addition, ores of certain metals such as aluminium, not yet found in Canada, are now imported for manufacture and export owing to the abundance of cheap hydro-electric power. There are seven copper smelters in Canada feeding three refineries, the capacity of which is ten times the annual domestic consumption of copper in the Dominion, being equal to the demand from the whole of the British Empire. Lead and zinc are treated at the Tadanac smelter at Trail, B.C., in one of the largest refineries in the world, whilst a large tonnage of zinc is also made at Flin Flon, Manitoba. Nickel is supplied to practically the whole world from the mines and refineries in Ontario, whilst cobalt metal is still furnished to the extent of half the world's supply from the refinery at Deloro, Ontario.

Improvements in Extracting Zinc

REMARKABLE improvements in the Maier process for the reduction of zinc ores by the use of natural gas were announced at the meeting of the American Zinc Institute held in St. Louis, on April 18. These improvements, which it is believed furnish the final steps in making this radically new zinc process successful, are the result of a year's further experimentation by the United States Bureau of Mines at its Reno, Nevada, and Berkeley experiment stations, following the announcement of the discovery of the process at the Institute's meting a year ago. The difficulties encountered in large scale experimentation on the process have been overcome by the use of a catalyst which is placed in the retort just before the gases enter the condenser. This catalyst, whose composition has not been fully revealed, causes the complete elimination of oxidising gases even when the process is operated at higher temperatures and rates than heretofore possible. The essential features of the process as it has been so far developed by the Bureau, and which it is now hoped will be further developed by industry comprise the continuous downward flow of calcine through a metal retort at temperatures of 1050-1100°C, concurrent flow of natural gas to secure intimate contact with calcine and treatment of gases resulting from reduction with excess methane in the presence of a special catalyst. The fuel-reagent expense of the natural gas process of zinc reduction represents the equivalent of coal at $1.20 or about 5s. per ton, which is of course a substantial lessening of the important item of fuel costs. The development of the Maier process is the outcome of fundamental studies of metallic oxides begun by the Bureau of Mines some years ago. The success of the experimental work is largly due to the application of precise knowledge of chemical fundamentals, a field in which the Bureau's Berkeley station has specialised. Costs of reagents, thermal economies, maintenance, labour and recovery of by-products, together with cost of equipment are all favourably influenced by the new process, which is capable of yielding metallic zinc of more than 99.9 per cent. purity.

The Outlook for Tin

DEALING with the outlook for tin at the meeting of the London Tin Corporation, held on April 28, Mr. John Howeson, the chairman, was quite optimistic, but emphasised that the world needed higher commodity prices. The maintenance of pre-war prices to which they had now reverted in a post-war world of debased currencies, a world strangled with debt and held back by needless unemployment, was clearly a social and an economic impossibility. The producers of tin, whose house was perhaps, the least disordered of all primary producers, were now ready to lead the way, undismayed by the cost, which was represented by the need of regulating their output to whatever basis consumers might determine. The complete plans under consideration were comprehensive and adequate, and they had the cordial backing of the industry. Mr. Howeson concluded by predicting that the worst had been seen so far as tin was concerned. With output completely under control and the industry more solid than ever before, the producer might look with certainty in the not far distant future for that compensation to which he was entitled by his courage and the sacrifices he had made in the common cause.

American Electro-Chemical Society

A LARGE number of papers dealing for the most part with various problems connected with the electro-deposition of metals was presented at the annual meeting of the Electro-Chemical Society held in

Baltimore, from April 21 to 23. Of the remainder which dealt with the other aspects of electro-chemistry and electro-metallurgy, one by Professor Alfred Stock calls for special mention. It is devoted to the subject of beryllium, and sums up most of what is known about this metal.

The first part of Professor Stock's paper deals with the history of the metal, which was first prepared in the pure state by Lebean, in 1898. From 1919 to the present time Professor Stock has, himself, been very closely associated with Dr. Hans Goldschmidt, Dr. Paul Praetorius and Dr. Otto Preiss in developing, under the auspices of Siemens and Halske, a technical method for the industrial preparation of the metal. That method involves sintering beryl with sodium silicofluoride, which is dissolved in water, associated iron being eliminated by blowing air through the solution, adding caustic lime and thus precipitating calcium fluoride and beryllium hydroxide. Evaporation yields a beryllium oxyfluoride containing from 20 to 23 per cent. of beryllium which dissolves readily in fused beryllium double fluoride, which forms the electrolyte for the subsequent electrolysis of the metal.

Extraction of Beryllium Metal

THE fusion mixture described above is now being substituted with great success for the barium beryllium fluoride formerly employed. The temperature at which electrolysis is effected is 1400°C. Acheson graphite crucibles are employed and the heating is carried out by packing the crucible with coke and passing current, by means of a graphite electrode, through the mass. The operation of a cell takes from 18 to 24 hours; 90 per cent. of the beryllium in the fused salts is recovered and a metal containing no more than 0.3 per cent. iron, 0.07 per cent. aluminium and 0.04 per cent. carbon, and the remainder beryllium is obtained. Other methods used by other investigators are described. The density of the beryllium so obtained is 1.83, and the melting point 1278°±64°. The paper next describes its uses, and gives a useful warning that, on acount of its high cost and its brittleness, beryllium is not likely to become a metal for general use, and that statements in the press regarding its employment in aeronautical construction "must be regarded as fantastic." Its transparency to X-rays, nearly twenty times that of aluminium, makes it very useful for windows in X-ray tubes. It can be electro-deposited on copper, nickel, iron, aluminium, and alloys of the light metals, and the subsequent heat treatment of the coating enables an alloy to be formed with the base. Its greatest potentialities appear to lie in the great hardness it imparts to copper, and the high electrical conductivity of beryllium bronzes.

Weathering of Tinned Sheet Copper

ACCELERATED weathering tests of tin and solder-covered copper are usually made in humid atmospheres containing 1 per cent. SO_2, 5 per cent. CO_2, and 94 per cent. of air by volume, maintained at a temperature of 50°C. or over. Recent research at the United States Bureau of Standards has shown that tin and electro-tinned coper are but slightly attacked by this atmosphere. On the other hand, heat-treated electro-tinned copper and hand-tinned copper are corroded and very badly pitted, while soldered copper, if heated to high temperatures previous to weathering, is markedly furrowed, resulting in a decrease not only of the strength of the sheet at the joint but also of the number of bends required to produce failure. The pitting is due to the formation of a brittle copper-tin layer which, upon cracking, exposes the copper to form the cathode of a galvanic cell with it. The furrowing of copper at the solder-copper junction is also due to similar galvanic action. Microscopic study revealed that the copper alloyed with the molten tin of the tin coating or of the solder to form definite layers. The layers of tin-copper alloys adjacent to the sheet became richer in copper the higher the temperature used during tinning or soldering. The pitting of copper caused by galvanic action set up between these copper-rich alloys was considerable. At lower temperatures, less alloying occurred, with the result that the tin coat contained very little copper and the pitting and furrowing were markedly diminished. Poor technique in the use of flux is not the cause of seam corrosion, but can cause pitting of copper. The prevention of seam corrosion and pitting of copper coated with tin-bearing materials lies in the prevention of the formation of copper-tin alloys.

Aluminium Bronzes

THE so-called aluminium bronzes have a wide range of uses according to their composition, and in many cases heat treatment produces a great difference in properties. From the binary diagram for aluminium and copper it might be expected that any alloy with over $7\frac{1}{2}$ per cent. aluminium should be hardenable, but in practice it is found that for noticeable results the aluminium content should exceed $8\frac{1}{2}$ per cent. Alloys containing less than this are soft and ductile and suitable for cold working, whereas those containing a little more aluminium are capable of being hardened by quenching. Even the bronzes with aluminium as high as 9 or 10 per cent. may be worked both hot and cold, and severe cold work may produce in small sections tensile strengths as high as 150,000 lb. per square inch, with, of course, decreased ductility. Iron has an especially marked effect, giving a finer grain size and greater strength on cold working, which strength may be largely retained on later re-heating. Due possibly to the finer grain, it also has a tendency to increase corrosion resistance. The iron is commonly used in amounts of approximately 3 per cent., and in American practice it is used more than other elements. In foundry operations a number of special precautions must be taken. It is important to keep the metal free from contact wih reducing gases, which are readily absorbed by the metal. The shrinkage during solidification is high, and therefore tends to produce pronounced pipe; this has to be provided for by the use of large heads of risers.

Aluminium oxide dross forms readily and may cause a dirty surface for some depth if pouring is not carefully done. Precautions should be taken to avoid agitation of the metal, as this drossy surface causes trouble with cutting tools. Phosphorus is injurious and should not be used as a deoxidiser. In some cases the addition of small amounts of lead is desirable, as it improves machining qualities, with only a moderate reduction in tensile strength.

Gas in the Metallurgical Industry

The Work of the Birmingham Industrial Research Laboratories

In a little over twenty years the Birmingham Industrial Research Laboratories have grown to their present size from one small laboratory and a test foundry. The technical staff was originally six but it is now forty, comprising engineers, physicists, chemists, heat treatment experts and furnace experts, and whilst their ultimate aim is to increase the industrial gas load they have now become a communal research laboratory not only for the gas industry, but for every industry using heat.

In the course of their evolution, the function of these laboratories has widened enormously from their original object, which was to increase the gas load by helping the industrialist to use gas in the right way. Experimental work on gas apparatus has led to experimental work on the products of the apparatus. For instance, research on lead melting furnaces for making accumulator plates gave rise to three years of very illuminating research on accumulators in general. Work on metal melting and heat treatment necessitated metallurgical experiments and testing machines for strength of materials, so that now the department has chemical and metallurgical laboratories and testing machines for loads up to a hundred tons.

General view of the Birmingham Physical Testing Laboratory, where the testing of ferrous and non-ferrous materials is carried out for manufacturers

Much of the recent experimental work of these laboratories has been devoted to the thermostatic and automatic control of the higher ranges of temperature required in industrial processes. A typical example is that of some successful research carried out over a period of two years on furnaces used for case hardening in the shops of the Moss Gear Co., Ltd., Tyburn. The primary object of the research work was to ascertain how far improvements in working efficiencies could be effected in a battery of four town gas-fired furnaces already in use. The lines of research involved an examination of such matters as insulating material, improved recuperative elements, the precise control of primary and secondary air, and reduction of losses by leakages through doors and dampers.

Part of the Heat Treatment Section of the City of Birmingham Industrial Research Laboratories, showing gas furnaces and gas-heated salt baths for demonstration purposes and for carrying out research work

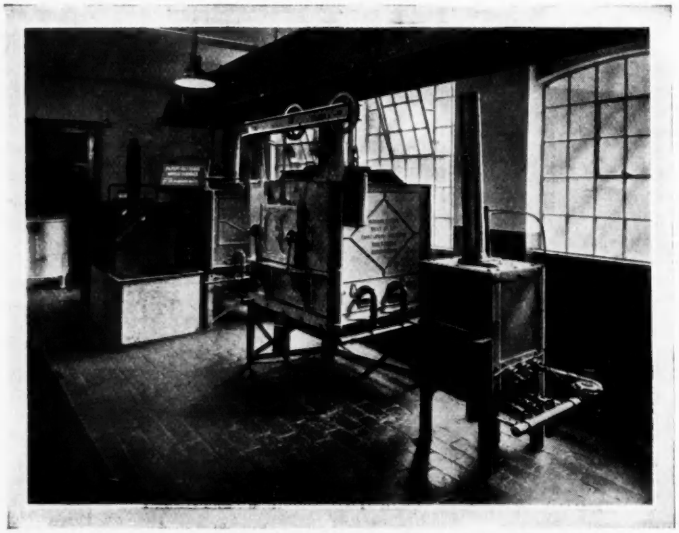
Two of the latest forms of large gas fired oven furnaces as used for case hardening operations and for the heat treatment of large dies

The first test, carried out over a period of a working week, consisted of the heat treatment of mild steel motor gear parts, placed in suitable carburising compound, in either welded wrought iron, mild steel plate or nickel chrome boxes. The control of gas to the furnace, and also of the primary and secondary air and waste gases, was carried out by hand. From a summary of this test it was shown that the throughput in lb. per hour was 113 and that the consumption of gas per lb. of material (gross) was 3.72 cu. ft. A second test on similar lines on another furnace of the same type gave a throughput per hour of 102.7 lb. and a gas consumption per lb. of material of 3.57 cu. ft.

The third test, on the same furnace as that used for the second test, was made after the furnace had been equipped with a special automatic gas control apparatus, the primary and secondary air supplies being fixed to suit the consumption. The control was on what is called the "hit and miss" principle, that is to say, the gas supply was either full on or shut down during the working period. A safety device was fitted whereby the gas supply would be automatically shut off if the thermo-couple failed or burned out from any cause. A time switch was also provided by means of which the gas valve could be automatically opened at any predetermined time, so lighting up the furnace without the need of an operator. This device is of particular value after the week-end shut-down. As the outcome of these improvements in design, the third test gave the following improved results: A throughput in lb. per hour of 141, and a consumption of gas per lb. of material (gross) of 2.87 cu. ft.

The fourth test, in the same furnace as that used for test 3, was made after the automatic control of gas supplies and the damper of the furnace had been synchronised. Full details of this control cannot be given here but the results of the test, carried out on the lines of the preceding ones, show that the throughput of the furnace in lb. per hour had again been increased to 156.8 and that the consumption of gas per lb. of material (gross) had been still further reduced to 2.78 cu. ft., as against the 102.7 lb. and 3.57 cu. ft. of the second test.

Comparison of these improved results with the best results obtained from gas furnaces in 1924 shows that a reduction of over 60 per cent. has been effected in the fuel consumption for a given amount of work.



A corner of one of the three Industrial Demonstration Rooms at Edmund Street, Birmingham, where gas fired furnaces can be seen under working conditions by manufacturers

During the course of these tests an electrically-heated furnace was also installed, and records were taken of the results obtained from it. These were, of course, compared with the results obtained from the gas furnaces. Electricity was supplied at a very low cost per unit, and at the time the final tests on the gas furnace were taken the cost of gas for a given amount of work was found to be about 30 per cent. less than the cost of electricity. The one electrical furnace is still retained by the Moss Gear Co. for carburising operations, but there are now six gas fired furnaces in use for the same purpose.

Four gas heated plating tanks in the Birmingham Art Metal Works, placed in brick linings, with the result that a 33⅓ per cent. saving has been effected in gas consumption and output has been augmented

Heat Treatment of Metals

The most important application of town's gas to industries in Birmingham is the heat treatment of metal, in particular steel and brass. Brass, copper and cartridge metals are used extensively in the manufacture of stampings for all classes of work, from door furniture to motor car lamps and fittings. After each process of cold working, such as blanking, pressing and forcing through dies, these materials are annealed by heating up in gasfired furnaces

to a temperature of about 700° C. In some instances a brass pressing may undergo many annealings before it assumes its final form, the annealing taking place between each process of drawing and stamping. This method of restoring softness to the material is also applied to precious metals in the many large and small jewellery manufacturers' shops in Birmingham and district. Sheet silver, now used so extensively in the manufacture of brush backs and flower vases, is annealed in gas furnaces between cold working (such as stamping, pressing and spinning) to restore the ductility of the metal.

For annealing steel and other ferrous material the temperatures required range up to 900° C., but are easily attained and maintained in the latest forms of gas furnace. The normalising of steel parts is a process somewhat similar to annealing, except that the rate of cooling of the material after it has been raised to the required temperature is accelerated by allowing the material to cool off in cold air instead of in the interior of the heated furnace. Many millions of cu. ft. of gas are consumed annually for this process. The work—on trays, or in the form of coils or sheets—is charged into the muffles or ovens. After it has undergone the process of normalising it is then formed into the final product. In the hardening and tempering of steel—such as is necessary for tools, knife blades and other articles with a hard cutting edge—steel of high carbon content is used, as it has the property of becoming hard when quenched in water from a suitable temperature. But knife blades, sewing needles, hosiery needles, knitting pins, fish hooks, razor blades and so on cannot be left in this condition as they would be too brittle and would break in use. To overcome this brittleness an after process known as tempering is necessary. This gives the extremely hard and brittle steel the essential property of elasticity.

In all these process, gas is used very extensively as a heating medium. For small articles which must be kept free from scale special furnaces are used with gas and water seals which prevent any contact with the air during operation. The gas industry, despite prevailing trade depressions and many antiquated and unfair legislative handicaps, advances consistently year by year. The extent of the progress made will be realised when it is known that the annual increase in the quantity of gas sold in this country, converted to a basis of heating value, has been consistently maintained at nearly twice that of the corresponding increase in the sales of public electricity.

Nickel-Steel Direct from Ores

Commercial Possibilities for New Process

THE Mines Branch of the Dominion Department of Mines at Ottawa has just issued a bulletin by T. W. Hardy and H. H. Bleakney entitled "The Semi-Direct Production of Nickel Steel from Sudbury Ore." As a result of the research work conducted at the Government laboratories it appears that there are possibilities for commercial production of nickel steel direct from the nickel-copper ores of the Sudbury district of Ontario. A nickel-iron sponge has been made successfully by a direct process from the ore, and the nickel steel production from this sponge is stated to compare favourably in physical or strength tests with nickel steels of similar specifications made in the ordinary way.

The metallurgical practice at present followed in the treatment of the Canadian nickel ores isolates nickel in pure form while the larger content of iron in the ore is wasted as slag.

A Patented Process

The fact that these ores contain the iron and the nickel so laboriously separated and that iron and nickel are afterwards re-united in the manufacture of nickel-irons and steels has been the incentive for several attempts to produce nickel-iron alloys directly from the ores. The Colvocoresses process, developed and patented by G. M. Colvocoresses, is the one generally referred to as the most interesting of such attempts, as it produced a nickel-copper pig iron containing nickel, copper and iron in the same proportions as they existed in the ore. It is believed that the large proportion of copper, which was as much as one-third of the nickel content, prejudiced the steel trade and the process involved large capital expenditures including both blast-furnace and open-hearth plants.

By the new process, developed in the Mines Department laboratories, much circuitous procedure would be eliminated and the iron which occurs in large proportions in the ores would be utilised rather than wasted in the slag. It is stated that none of the steps are difficult and most of them are already in practical operation in other processes. Concentration by selective flotation of disseminated ore produced copper concentrates and also the nickel-iron concentrates which by successive stages in roasting, sintering and other heat treatments were reduced to the metallic nickel-iron sponge ultimately turned out in the form of briquettes. These briquettes were used in practical tests in the making of nickel-steel ingots, the charges to the furnace including, besides the briquettes, suitable weights of boiler-plate punchings along with ferro-alloys necessary to produce the specification of steel desired. Steels made by the process compare favourably with steels made by methods commonly used. There are good reasons for believing that the process has commercial possibilities.

British Empire Mineral Resources

Detailed Economic Survey now in Progress

IN view of the assembly at Ottawa of the Imperial Economic Conference, a paper recently discussed at the annual meeting of the Canadian Institute of Mining and Metallurgy is now of particular interest. This paper, which can be consulted at Canada House, Trafalgar Square, S.W.1, has been prepared by Dr. Charles Camsell, Deputy-Minister of Mines of Canada, and discusses "The Mineral Position of the British Empire," following on similar discussions which have taken place at the Empire Mining and Metallurgical Congresses held in Canada in 1927 and in South Africa in 1930. It is pointed out that the British Empire stands in an unrivalled position with regard to supplies of minerals and metals, but the trade channels in minerals and products thereof lead largely to and from foreign countries. Dr. Camsell stresses the important part that minerals in raw and manufactured form play in international trade, and the great scope for development of inter-Empire trade in minerals by the exchange of surpluses found in one part of the Empire for deficiencies in others. What is needed more than anything else in order to illuminate the existing situation in the British Empire, from the point of view of mineral production and exchange, is a consolidated statement or balance sheet showing at a glance the net surplus or deficiency of the Empire with respect to each important mineral. This task is now being attempted as the result of a recommendation made by the Third Empire Mining and Metallurgical Congress held in South Africa in 1930. The plan, when completed, will indicate the surpluses and deficiencies of each unit of the Empire and will also show the line upon which inter-Empire trade may be developed.

Canada as a Mineral Producer

The British Empire mineral position may be regarded as having unique strength. Of 28 important metals and minerals, the Empire possesses 21 in such abundance as to have large quantities available for export. Of the remaining 7 minerals, the Empire possesses 2 in quantities adequate for its own needs; for one the Empire is partially dependent on foreign sources; and for 4 it is almost entirely dependent on foreign sources. No other country, or group of countries politically associated, occupies a position comparable in respect of supplies of mineral raw materials. The strength in mineral resources, however, depends entirely upon the Empire being regarded as one unit, in the combined resources of all and not in those of any one country. Maintaining that the position in Empire mineral trade is one of incalculable possibilities, Dr. Camsell quoted figures showing that Canada alone in the decade 1921 to 1930 inclusive transacted an aggregate external business in minerals and mineral products reaching the sum of approximately £1,250,000.

Some Inventions of the Month

BY OUR PATENTS CORRESPONDENT.

Agglomerating Ores

A DAMP mixture of finely divided ore, particularly zinc ore, and coking coal is superficially ignited on a support permeable to gases, such as a Dwight Lloyd sintering apparatus, and the charge is subjected to an up or down blast to produce an ore coke. The ignition may be effected by a stationary or travelling furnace or by hot gases. Air low in oxygen, *e.g.*, the hot waste gas from the process itself, is used. Thus air may be passed through a layer of material such as coal to lower its oxygen content before it reaches the charge. The charge may be sprayed with water to prevent incipient combustion of the coke. See Specification 362,796, International Convention date April 2, 1930, by Metallges. A.-G., of Frankfort-on-Main, Germany.

Beryllium Alloys

TO obtain alloys of beryllium with iron, copper, nickel or other heavy base metals, the beryllium is liberated from an easily fusible slag, containing in addition to beryllium oxide at least one alkaline-earth metal oxide with or without an addition of one or more alkali metal oxides, by means of a reducing agent in the presence of one or more fused heavy metals. Specified reducing agents are carbon, silicon, aluminium and carbides. The compounds of beryllium and of the alkali and alkaline earth metals may be such as are converted into the oxides at the reaction temperature, *e.g.*, carbonates. The alloys may be freed from residual reducing agent or other impurities by reaction with a further quantity of the slag. See Specification 363,837 (Baron), dated December 23, 1930, a communication from Siemens and Halske A.-G. of Berlin.

ALLOYS containing beryllium and other alkaline-earth metal are made by adding these to iron or copper or alloys thereof, excepting copper-nickel alloys, so that the total content of alkaline-earth metal is 0.2-15 per cent. The other metals present may be nickel, cobalt, chromium, titanium, vanadium, molybdenum, tungsten and zirconium with or without approximately 1 per cent. of carbon, silicon, or phosphorus. The alloys may be aged at a comparatively low temperature, say 300° C., after being cooled quickly from at least 50° C. below the melting point. See Specification 363,861, International Convention date January 21, 1930, by Siemens and Halske A.-G., of Berlin.

Coating with Metals

A PROCESS for coating iron or steel articles with aluminium or its alloys comprises the steps of (a) removing fatty substances, oxides and slags, *e.g.*, mechanically or by pickling, (b) removing superficial impurities by immersion in an aqueous solution or a melt of a salt, such as zinc ammonium chloride or zinc ammonium fluoride, which will convert the oxidic impurities into volatile compounds and, when the articles are dried by heating, will leave thereon a protective salt layer, (c) dipping the articles in a molten flux bath containing zinc chloride and an alkali chloride, calcium chloride and an alkali ammonium fluoride, and (d) transferring them to the metal bath without allowing their temperature to fall below the melting point of the metal. In the flux bath the articles are preferably kept in movement or subjected to brushing. See Specification 363,158, dated January 5, 1931, by E. H. E. Johansson, of Stockholm, Sweden.

Concentrating Ores

A FROTH flotation process described in Specification 362,927, International Convention date September 10, 1929, by Minerals Separation, Ltd., of London, assignees of C. H. Keller of San Fransisco, U.S.A., comprises the use as flotation agent of a xanthic derivative of a substance containing in its molecule a closed chain of five atoms, of which four are carbon, *e.g.*, a furane or thiophene. Furfuryl xanthate is referred to as a suitable agent and examples are given of the treatment of various copper, zinc and lead ores.

IN a froth flotation process for concentrating ores, especially oxidised ores, described in Specification 363,122, dated December 6, 1930, by Minerals Separation, Ltd., of London, flotation agents are employed conjointly, *viz.*, (1) an organic thio derivative, *e.g.*, a xanthate or dithiocarbamate, (2) a fatty acid, *e.g.*, oleic or other higher fatty acid or an alkali salt thereof, and (3) a deflocculating agent such as sodium silicate or silicic acid sol. The thio derivative may be any organic compound in which carbon is linked to sulphur by a double bond with or without a further sulphur atom linked to the carbon atom. Examples relate to the treatment of an oxidised copper ore containing copper silicate, with the use as flotation agents of potassium ethyl xanthate, oleic acid and sodium silicate, and to the treatment of a cassiterite ore with the use as flotation agents of potassium ethyl xanthate, oleic acid and soda ash.

THE concentration of oxidised copper ores or of mixed oxidised and sulphide copper ores by froth flotation is facilitated by addition of ammonium cyanide or hydrocyanic acid, either by grinding with the ore or by addition to the pulp in the flotation cell. The flotation may be effected in stages, relatively little cyanide or none at all being used in the first stage. See Specification 362,961, dated September 3, 1930, by R. J. Lemon, of Norton-on-Tees and Imperial Chemical Industries, Ltd., of London.

Lead Alloys

ELECTRIC cable sheaths are made of an alloy produced by alloying pure lead with antimony and adding either (a) a chemical compound of antimony with one or more of the metals cadmium, copper, nickel, manganese or magnesium, or (b) a solid solution of antimony and tin. A suitable alloy may contain, for example, 98 per cent. or more of lead, up to 1 per cent. of antimony, and the remainder the third component. See Specification 362,973, International Convention date September 27, 1929, by Siemens-Schuckertwerke A.-G., of Berlin.

Steel Alloys

STEEL alloys are produced by the reaction of iron or an iron alloy with at least one oxide of an alloying metal or non-metal, *e.g.*, tungsten, silicon, manganese, chromium or nickel, the materials reacting in the liquid state under reduced pressure and in presence of free or combined carbon and of an addition of one or more of the elements nickel, silicon, phosphorus, aluminium or titanium. In one example, a stainless steel made from soft ingot iron, metallic nickel, graphite and chrome ore contains 8.9 per cent. of nickel, 18 per cent. of chromium, 0.61 per cent. of silicon, and 0.016 per cent. of carbon. See Specification 362,823 (Every-Clayton), dated April 27, 1931, a communication from F. Krupp A.-G., of Essen, Germany.

International Nickel Company

This Year's Prospects

IN his address to the shareholders of the International Nickel Co. of Canada, on March 29, Mr. Robert C. Stanley, the president of the company, said that while operating expenditure has been reduced by 51 per cent., compared with 1929, the budget for research, sales and development has been increased by 25 per cent. over that of 1929. Even so, the sales policy has not been able to maintain nickel consumption at an adequate volume. Sales of nickel, including those by the Mond Nickel Co., Ltd., of 55,740,000 lb. compare with 27,380,000 lb. in 1921, also a year of serious business depression. From this gratifying comparison it may be inferred that the company's policy of technical sales development is showing concrete results. In the matter of platinum metals, improvement has had to wait upon group recognition of the industry's common interest. Late in 1931, as a result of months of discussions and negotiations, Consolidated Platinums, Ltd., was organised as an English corporation which will not only sell platinum originating in Canada, Russia, South Africa, and South America, but will also conduct an active campaign to promote and increase its uses. The company's great resources of rich ore, the extensive hydro-electric power development, and the modern plants for smelting and refining nickel, monel metal, copper and its precious metals remain unimpaired.

Metallurgical Section

June 4, 1932.

Current Topics

A Pioneer of the Basic Bessemer Process

DR. ARTHUR COOPER, who was one of Tees-side's leading pioneers in the iron and steel industry, died on May 24 at the age of 83. For the last year he had been in poor health and resided at Nantwich, Cheshire. In the development of the basic Bessemer process Dr. Cooper became closely associated with Sidney Thomas and Percy Gilchrist, and with these two metallurgists he filled an important part in the exploitation of this process, being awarded the Bessemer gold medal by the Iron and Steel Institute. In 1881 the North-Eastern Steelworks was formed, specially to work the Bessemer process, by Thomas, Gilchrist and Riley, who were associated with the late Sir Arthur Dorman. Dr. Cooper was then appointed general manager and later managing director of the company and when the works were merged with Dorman Long and Co., he became a director of that firm. He was associated with the Tees Conservancy Commission, whom he served for 25 years; was a past president of the Iron and Steel Institute, and a magistrate of the borough of Middlesbrough.

Developments in Industrial Steels

CHEMICAL engineering and metallurgical technique involve many processes in which temperatures between 600° and 1200°C. are attained and the durability and life of plant within this range of temperature is of considerable importance. In the past, high temperatures were met to a large extent by the use of refractory materials, but modern developments have demanded properties which cannot be obtained from even the most refractory non-metallic materials, and alloy steels and cast irons have had to be produced in order to solve the problems which arise. The extended use of such materials has been brought about by greater strength, toughness, and resistance to shock, and by the fact that not only can they be fabricated better, but also that they are more impermeable to gases, liquors, and molten salts. In the development of metals for high temperature conditions, the most important properties which they must possess are ability to withstand prolonged stresses at those temperatures, and resistance to oxidation, scaling, and chemical attack. A considerable amount of research work has been carried out during recent years with these conditions in view, and has resulted in the development of new materials for service in various industrial processes, especially in high pressure steam plant. Research work which has been carried out has dealt with the prolonged loading of metals at temperatures from 600° to 1000°C., and the amount of flow or creep which takes place at those high temperatures, together with the resistance of such materials to oxidation and corrosion at similar temperatures. The practical results of such researches have been the manufacture of steels of the high-chromium high-nickel type, with the addition of other elements such as molybdenum, silicon, tungsten, and vanadium, either together or alone, or of high-chromium high-nickel alloys in which little or no iron is present.

Anticipated Effect of U.S. Tariff on Copper

THE tariff of 4 cents per lb. (about £25 per ton) on copper imported into the United States was retained by the vote of the Senate at Washington on May 23, in accordance with the decision of the Finance Committee, and is expected to cause a revolution in the copper industry. Imports of refined copper into the United States for the first quarter of 1932 exceeded 50,000 tons, the whole of these shipments coming from Canada and Chile. The new duty will be more than sufficient to shut out all foreign metal, since the United States already produces more than it needs, and there is an estimated surplus stock of 700,000 tons on hand. It is feared, therefore, that an immediate effect of the tariff will be the disintegration of the World Association of Copper Producers, and all that it stands for, including the curtailment agreement, which has brought about an 80 per cent. reduction in potential output. The United States tariff policy, particularly in regard to copper, has been viewed with increasing concern in South America. The establishment of another European copper refinery, preferably in Great Britain, is suggested by the Chilean newspapers as a solution of the difficulties which will confront the Chilean copper industry in consequence of this action. The retention of the tariff is definitely resented in Chile, in view of the fact that copper forms a vital percentage of the country's exports.

Reduction of Iron Ore Sinter

As the use of iron ore concentrates increases and finer crushing becomes necessary to obtain a satisfactory recovery of iron, it is evident that agglomeration of this additional fine ore is essential to provide a satisfactory product for use in blast furnaces. Sintering has been adopted by the industry as the most satisfactory method of agglomeration, in consequence of which it has become important to investigate those properties of sinter which have a bearing upon its deoxidation or conversion to metallic iron in the blast furnace. It is now generally recognised that sinters

contain varying amounts of iron silicate formed during the sintering operation. One of the experiment stations of the United States Bureau of Mines, in co-operation with the University of Minnesota, has investigated the reducibility of iron silicate to determine the influence which this constituent of sinter has upon deoxidation. Particles of an iron silicate slag, ranging in size from "minus 3 to plus 14 mesh," were only about 35 per cent. reduced when exposed to carbon monoxide gas for 16 to 22 hours at 950°C. Under the same conditions such particles of several iron ores investigated were completely reduced in one hour. When "minus 80 mesh" particles of iron silicate slag were exposed to carbon monoxide for 17 hours at 950°C., 99 per cent. of the iron was present in the metallic form. The failure to reduce completely samples of several commercial sinters in laboratory tests has apparently been due to the presence of iron silicates.

New Uses Wanted for Titanium

ALTHOUGH titanium was discovered 141 years ago, it is only within the past 10 or 15 years that any extensive outlet for its ores has been found, chiefly due to the larger employment of titanium pigments. Ferro-alloys containing this element were largely used for a time, especially in connection with the manufacture of Bessemer steel rails, but as many railways adopted open-hearth rails in preference to Bessemer rails, the demand for the alloys in the rail manufacture gradually dwindled into insignificance. New outlets for ferro-titanium have been found in the steel industry, and the production of titanium alloys both in the United States and in Europe has increased in recent years. By far the largest use of titanium at the present time is for pigments. The manufacture of titanium white began in Norway, but the United States is now the leading producer. In its properties titanium closely resembles cerium, thorium and zirconium. In the amorphous form it is a dark grey powder, similar to reduced iron; the fused metal, resembling polished steel, is sufficiently hard to scratch quartz. Although the metal possesses little ductility it is readily worked when properly prepared, and at a red heat it can be forged and drawn. A number of alloys of titanium with other elements have been tested for different purposes, but the only alloys which have been recognised to any extent as articles of commerce are ferro-titanium, ferro-carbo-titanium, cupro-titanium and mangano-titanium. The quantity of titanium alloy added per ton of steel may vary from 1 to 13 lb.; its addition tends to produce a more homogeneous steel with freedom from blow holes.

Recovery of Precious Metals

Notable Increase in Flotation Practice

THE flotation of precious metal ores is on the increase. Many gold and silver flotation problems which defied solution in the early days of flotation practice, have now yielded to the attack of more highly developed chemical reagents and mechanical equipment. Flotation is not, however, to be considered a substitute for cyanidation, but rather as an accessory thereto. In all probability the combination of these two methods will extend the use of both, the flotation process being applied as a preliminary concentration treatment to raise the grade of the feed to the cyanide plant.

The problem of precious metal recovery has become especially important in the selective flotation of complex lead-zinc and copper-iron ores containing gold and silver. Before the advent of selective flotation, previous metal recovery was not given special attention in the treatment of mixed sulphide ores, the metallurgist being chiefly concerned with obtaining a high recovery of all the sulphides in a bulk concentrate. If the sulphide recoveries were high the precious metal recoveries were also high since both the free and sulphide associated gold and silver were recovered. To-day each valuable sulphide mineral is recovered in its respective concentrate, and the distribution of precious metals is watched with the same interest as that of the sulphides for the reason that smelter payments for precious metals depends on the base metal concentrate with which they are recovered.

Effect of Addition Agents

Addition agents, such as soda ash and lime, have a decided effect on the flotation of precious metals, particularly native gold. In connection with tests carried out at the Canadian Bureau of Mines, Ottawa, on copper-zinc ores containing gold it was observed that "the grade of (copper) concentrate produced was found to have an important bearing on the (gold) recovery, but for a given grade a higher recovery of gold was obtained with soda ash than with lime. These tests indicated that the best separation and recovery of gold with copper could be obtained by using a small amount of cyanide and floating the copper in a pulp made slightly alkaline with soda ash." Similarly, tests made at the American Cyanamid Co. ore dressing laboratory on an Australian copper-gold ore showed that lime exerts a strong depressing action on free gold.

Technical Information on Zinc

A New Circular of the U.S. Bureau of Standards

ZINC is now available commercially in a state of very high purity. Recent improvements in both the smelting process and the electrolytic process have made available metal with a zinc content close to 99.99 per cent., which has been found to be extremely resistant to corrosion by most of the common acids. On account of the relative ease with which zinc can be produced in large crystals, a great deal of study has been devoted to the properties of the metal in the form of single crystals and much light has been thrown on the subject of its plastic deformation. Zinc crystallises in the hexagonal system, and the basal hexagonal planes, that is, the planes perpendicular to the principal crystalline axis, are the ones upon which deformation by slip readily occurs. Although sheet zinc possesses very good tensile strength, it is not in the same class with sheet steel or iron. Zinc is much more ductile, and when maintained for long periods of time under sustained loading, it may "creep" or sag. This property of zinc must be borne in mind if it is to be used to best advantage in roofing and for other building purposes. By far the greatest outlet for zinc is in galvanising. In America nearly half of the zinc produced annually is used for this purpose. Zinc coating by galvanising, that is, dipping in molten zinc, is possible because of the ready alloying which occurs between liquid zinc and iron. This makes it possible for the metal to "wet" the iron surface, a circumstance not possible with some metals, for example, lead. Circular No. 395 of the United States Bureau of Standards which has just been issued, is devoted to zinc and its alloys. It contains a wealth of information on zinc, summarised from the technical literature and supplemented by the results of tests carried out at the Bureau.

Commercial Supplies of Indium

THE availability of indium in commercial quantities has been announced by William S. Murray, Inc., of Utica, New York. Ores rich in indium have been found and methods for the commercial extraction of the metal have been perfected and patented. Research has been successful in perfecting stable plating baths of the element and combinations with other elements. Indium has the effect of increasing surface stability and hardness of those elements to which it is added.

Blast Furnace Linings

The Chief Factors which Reduce Lining Life

THE results of investigations made during the past twelve years in the Department of Refractory Materials at Sheffield University in regard to the durability of blast furnace linings were referred to in a paper by W. J. Rees, read before the Refractory Materials Section, at the May meeting of the Ceramic Society. The chief factor operating to reduce lining life is stated to be direct corrosion of the brickwork in the hotter parts of the furnace by the slags normally present, but channelling or other abnormal local wear due to uneven distribution of the burden has occasionally been observed. Some attempts have been made to correlate slag viscosity with brick corrosion, but they have been indefinite in their results, and no connection between lining wear and the use of high calcium of dolomitic limestones as flux has been traced. Corrosion, and occasionally disintegration, of bricks from absorption of alkali salts is also important. Corrosion follows on alkali absorption in the melting zone and the lower part of the stack, whilst in the upper parts of the furnace alkali absorption tends to disintegration of the bricks and reduces their resistance to the mechanical shock imposed by the descending charge. Cases have been observed where from a high alkali content of the charge, crystals of alkali (mainly sodium) chloride and sulphate have grown in the bricks and joints in the upper parts of the stack and caused very severe disruption of the bricks. Deposition of iron chlorides in the brickwork also has tended to destroy the strength of the matrix of the bricks, but is less important in this direction than alkalies.

Disintegration due to Carbon deposition

Wear, due to mechanical weakness of the bricks, has been particularly noted in the stack, but it is likely that it has been an important factor in other parts of the furnace too. Although only one or two instances have been observed where disintegration from carbon deposition has been the definite cause of short lining life, this factor has appeared to be an infrequent cause of lining destruction. Occasional examples of wear have also been traced to unsatisfactory jointing cements, which have been used in rather thick joints and which have contracted and left openings. In this connection it has been noted that carbon deposition in joints is frequent and may be more serious than carbon deposition in the bricks themselves. Excessive carbon deposition in joints has been traced to the presence of metallic iron (presumably from the grinding plant) in the joint cement. Tests which have been made indicate generally that aluminous bricks are more resistant both to corrosion by slag and to corrosion by absorbed alkalies than siliceous bricks.

Whilst it has been evident that for the hotter portions of the furnace bricks of high refractory value are desirable, it has not always been noted that the bricks of highest refarctoriness have been most durable, owing to deficient mechanical strength and comparatively high permeability to slag and vapour. Some highly aluminous materials have proved less resistant to corrosion by the slag than moderately aluminous materials, and there have been definite indications that the presence in the brick of a sufficient amount of fluxing impurities to promote incipient vitrification at normal kiln temperatures (1,300° to 1,400° C.), so tending to mechanical strength and low permeability, is conducive to durability.

Cold Crushing Test

For the upper parts of the furnace the mechanical strength as measured by a cold crushing test has appeared to give indications of greater value than the test of "refractoriness under load." It has appeared likely that crushing tests at temperatures of 300° to 600° C. would give information of value. In experiments made with a view to observing any effect of chemical composition of the bricks on deposition of carbon within the brick in atmospheres of carbon monoxide, both dry and moist, at temperatures up to 1,000° C., the effect of ferruginous segregations in promoting dissociation of carbon monoxide, especially in the temperature range 300° to 500° C., has been noted, and in several examples it has been shown that reburning in an oxidising atmosphere at 1,300° to 1,400° C. considerably reduced the catalytic effect, and in one or two cases inhibited the carbon monoxide dissociation. From these experiments there is some suggestion that ferrous iron compounds are more likely to promote carbon deposition than ferric iron compounds.

Suggestions to Blast Furnace Managers

Resistance to slag attack is promoted by the use of well-burned bricks of good shape, low permeability, and good volume stability at high temperatures, made from aluminous clays with a refractoriness not below cone 30. The cement used should be finely ground, but contain a high proportion of grog, so as to avoid contraction on firing. Resistance to penetration and corrosion or disintegration by alkalies is promoted by the use of well-burned aluminous bricks of low permeability. Resistance to carbon deposition is promoted by the use of bricks of low permeability, well burned and free from ferruginous segregations. An iron oxide content not higher than 2.5 per cent. appears desirable, but the condition of the iron appears, within limits, to be more important than its proportion. Close jointing and the use of jointing cement free from metallic iron inclusions is also desirable. Resistance to mechanical wear is promoted by the use of bricks with a high cold crushing strength, whilst close texture and incipient vitrification appear to assist retention of resistance to mechanical shock and abrasion at the temperatures of the upper parts of the blast furnace.

United States Molybdenum Industry

Statistics for 1931

THE production of molybdenum concentrate in the United States during 1931 amounted to approximately 3,000 short tons, containing 5,221,000 lb. (average 85.93 per cent.) of molybdenum sulphide, MoS_2. In 1930 the production was 5,618 short tons of concentrates containing 6,205,000 lb. (average 85.75 per cent.) of molybdenum sulphide. The shipments of concentrates from mines in 1931 contained an equivalent of 3,157,000 lb. of elemental molybdenum valued, more or less arbitrarily, at about £345,000, as compared with shipments in 1930 of 3,759,269 lb. of molybdenum, valued at about £425,000.

Since by far the bulk of the production of molybdenum is used in steel manufacture, it follows that the drastic curtailment in steel mill operations was not without effect upon the consumption of molybdenum. However, although the production or molybdenum-treated steels undoubtedly showed an absolute drop in 1931, there continued to be a relative increase in the production of such steels as compared with the production of plain carbon and other alloy steels. Molybdenum is used chiefly in combination with other alloys, especially chromium, nickel and vanadium. These steels are used extensively in the automotive industries and a new group of nickel-molybdenum steels are finding applications in the chemical industry because they are able to resist hydrochloric acid as well as sulphuric acid. Nickel-molybdenum alloys with chromium added will also resist nitric acid and wet chlorine but these alloys cannot be forged or rolled into sheets like the simple nickel-molybdenum steels. High molybdenum steels, up to 15 per cent., have been made and actually rolled commercially, but due to the high cost of the element and a tendency of molybdenum to volatilise, such steels have not been widely used. Molybdenum, however, is an important constituent of many tool and magnet steels and in the high-speed steels 1 per cent. of molybdenum tends to replace 2 to 2½ per cent. of tungsten. Possibly the principal commercial development during the year was the rapid increase in the use of molybdenum in cast iron.

Three companies produce all but 2 or 3 per cent. of the world's molybdenum. Two of these—the Climax Molybdenum Co., operating in Colorado, and the Molybdenum Corporation of America, operating in New Mexico—are in the United States, and one—The Norska Molybdenprodukter A/S—operates at Knaben, northeast of Stavenger, Norway.

Platinum Alloys in the Chemical Industries

By EDMUND R. THEWS

PRACTICALLY all the platinum metals, alloyed with each other or with gold and silver, are now employed for industrial purposes, their usefulness being based chiefly upon their high chemical resistances combined with valuable mechanical properties. The following article deals with the properties of various groups of alloys.

THE platinum group of metals, including platinum, iridium, osmium, palladium, rhodium, and ruthenium, whilst exhibiting certain properties characteristic of all of them, may be subdivided into three pairs, each of which differ from the others in certain individual chemical properties, *viz.*, platinum-palladium, iridium-rhodium, osmium-ruthenium. The chief chemical differences between the metals of these three basic groups, in their action towards mineral acids, are (1) platinum and palladium are the only two metals of the platinum group attacked by aqua regia; (2) palladium is the only metal easily dissolved by boiling concentrated nitric acid; and (3) only platinum and palladium are attacked by boiling sulphuric acid.

All the platinum metals as well as gold are highly resistant to the other chemical reagents at ordinary temperatures, although they differ considerably in their reactions with fused alkalies. Fused sodium peroxide is the most corrosive, and all of the noble metals are more or less attacked by this reagent, their resistance decreasing in the following order: Rhodium, iridium-osmium-ruthenium, gold-platinum-palladium. Fused sodium hydroxide attacks osmium strongly. Platinum, rhodium and ruthenium are somewhat corroded by this reagent, whilst palladium, iridium and gold are inert to its action. Fused sodium carbonate exerts a rather slight action on platinum, rhodium, and osmium, but does not touch gold and the other platinum metals. Fused potassium bisulphate strongly attacks rhodium; it is less corrosive towards platinum, palladium and osmium but without any action on iridium, ruthenium and gold. Fused alkali cyanides dissolve gold but exhibit only a slight corrosive action on platinum and palladium, being without action on the other platinum metals. Similar differences of properties are exhibited by the platinum metals towards the various halogens. Chlorine, iodine and fluorine attack iridium, osmium, rhodium and ruthenium but little; chlorine is strongly corrosive towards platinum and palladium at red heat, whilst palladium is strongly attacked by iodine, and platinum by fluorine, at elevated temperatures.

Platinum-Iridium Alloys

The alloys of these two metals are the most widely used of all the platinum metals. Their hardness increases with rising iridium content up to 40 per cent. iridium, decreasing gradually as this point is exceeded. One of the important technical advantages of these alloys is that this increase of hardness is not accompanied by a corresponding increase of brittleness, although certain impurities if present in one of the alloying constituents may cause rapid embrittling of these alloys. On heating the platinum-iridium alloys to temperatures of 1,650-1,900° F. their surface is turned black by oxidation. This colouration disappears at higher temperatures, not however by reduction of the oxide, but by volatilisation, the basis of the black oxide being the volatile iridium. Extended heating of these alloys at temperatures above 1,650° F. will therefore be accompanied by a gradual loss of weight; they are not, for this reason, as well suited for pyrometric purposes as the platinum-rhodium alloys which do not suffer any loss of weight at much higher temperatures.

The chemical resistance of the platinum-iridium alloys to the action of nitric acid, aqua regia and sulphuric acid increases with rising iridium contents. The 5 per cent. alloy is easily soluble in hot concentrated aqua regia, the 10 per cent. alloy is noticeably less soluble, the 20 per cent. mixture dissolves very slowly, whilst the alloy with 30 per cent. iridium is insoluble for all practical purposes. The resistance of the platinum-iridium alloys to boiling concentrated sulphuric acid also is very much more pronounced than that of the unalloyed platinum. Experiments carried out at the Heræus plant indicated that if the loss of weight of pure platinum suffered by 40 days of boiling in concentrated sulphuric acid is assigned the value 100, the relative loss of a 5 per cent. platinum-iridium alloy is 73, and that of a 10 per cent. alloy, 58 (Reinglass, "Chemische Technologie der Legierungen," p. 433). These alloys, however, possess the serious disadvantage that on continued employment in connection with boiling sulphuric acid they gradually become brittle.

Of considerable importance are the relatively high strength values of technical platinum-iridium alloys employed in the chemical industries. Hardness as well as tensile strength increases with rising iridium contents up to 35 per cent. A peculiar characteristic of the platinum-iridium alloys rich in iridium is the singular effect of annealing at various temperatures on the strength and softness of the metal. Whilst with rising annealing temperatures the softness of pure platinum and of the lesser platinum-iridium alloys (up to 10 per cent. iridium) is gradually decreased, the iridium-rich alloys exhibit constantly decreasing softness values and increasing tensile strengths with rising annealing temperatures up to 1,400° F. On being subjected to higher annealing temperatures the strength values of these alloys exhibit a downward tendency. The substitution of iridium in these alloys by cheaper metals is rarely practised, the chemical resistance of all such alloys being lower than that of pure platinum. The best results may, perhaps, be obtained with platinum-gold-palladium alloys. Binary platinum-gold alloys are hard but brittle, whilst palladium alone exerts only a limited hardening effect.

Platinum-Palladium Alloys

The platinum-palladium alloys, including the palladium-rich mixtures, are much softer than the other platinum alloys employed in the chemical industries. The two metals are miscible in all proportions and excel in workability. On being alloyed with platinum, palladium loses practically all of its individual characteristics. For instance, whilst pure palladium is easily soluble in boiling nitric acid, the platinum alloy with 25 per cent. palladium is practically insoluble in this reagent. Also the colouration exhibited by pure palladium on heating to elevated temperatures does not appear on the 25 per cent. platinum alloy. Since the oxides of both platinum and palladium volatilise at elevated temperatures, their alloys may be heated to any industrial temperature without suffering noticeable loss of weight. The physical properties of platinum are not changed by the addition of palladium as much as by that of any of the other platinum metals. The hardness values of these alloys do not nearly reach those of the corresponding platinum-iridium alloys. Increasing gradually, the hardness of a 30 per cent. alloy is 180° Brinell (105° Brinell for the annealed alloy); beyond this percentage, the hardness values of these alloys are gradually lowered. However, the frequent assertion that certain platinum-palladium alloys are softer than pure platinum is erroneous, the hardness figures for hard and annealed platinum being 97° and 47° Brinell respectively.

On account of their lack of hardness, binary platinum-palladium alloys are rarely employed for construction work in the chemical industries, much better mechanical properties being obtained with the platinum-gold-palladium alloys or even with the binary palladium-gold alloys. A proprietary alloy "Rhotanium," containing, besides gold, 10-40 per cent. of palladium has been recommended as a substitute for platinum for electrical and dental purposes as well as for certain chemical purposes; these alloys are claimed to be very resistant to hydrochloric, hydrofluoric and sulphuric acids and fused soda, and to be easily rolled and welded.

Platinum-Rhodium Alloys

The platinum-rhodium alloys, whilst exhibiting remarkable mechanical and chemical properties, are not being employed extensively, a fact entirely due to the small quantities of rhodium produced. In its hardening effects on platinum, rhodium stands midway between iridium and the weak hardening agent palladium, whilst its reaction towards atmospheric oxygen is similar to that possessed by platinum; the very thin film of oxide produced at elevated temperatures is completely

reduced back to metal at a somewhat higher temperature. Rhodium oxide is not volatile at ordinary temperatures. These platinum-rhodium alloys are quite useful for a variety of purposes, one of its most well known applications being in the resistance wires of the Le Chatelier pyrometer couples (10 per cent. rhodium). Compared with the platinum-iridium couples the 10 per cent. rhodium alloy possesses the considerable advantage of remaining constant at temperatures as high as 2,900° F.

Platinum-rhodium alloys are also employed in the modern nitric acid contact process in which an ammonia-air mixture (10: 1) is passed through platinum-rhodium gauze serving as catalysing agent. In replacing the platinum gauze formerly employed almost exclusively, the platinum-rhodium gauze exhibits a contact efficiency of almost 100 per cent. In America, 3.5 per cent. platinum-rhodium alloy is being employed in the production of laboratory crucibles and dishes, the rhodium serving to improve the hardness and tensile strength of the platinum; the resistance of this alloy to the action of corrosive liquids and fluxes is considerably higher than that of pure platinum, while the very low degree of volatility of platinum is still further reduced by rhodium. The latter property of rhodium has caused this metal to be extensively employed in the production of 10 per cent. platinum-rhodium alloys for resistance wires in electrical furnaces, particularly where the furnace temperatures exceed 2,000° F. Apart from their high resistance to elevated temperatures, these wires exhibit a high coefficient of electrical resistance and a lower speed of crystallisation than platinum, so that in spite of higher initial costs they are frequently preferred to platinum wires in electric furnace construction.

In platinum-osmium alloys there is an exceedingly powerful hardening effect exerted by the osmium on the platinum, but in view of the high degree of volatility of osmium oxide at comparatively low temperatures, and since the osmium forms oxide even if present in the alloyed state, this property actually prevents any extensive use of the metal and its platinum alloys. The poisonous effects produced in the human system by osmium oxide resembles somewhat the indications produced by arsenic and cadmium vapours, besides causing serious affections of the eyes which in a number of cases have led to blindness. The general properties of platinum-ruthenium alloys exhibit a strong resemblance to those of the respective osmium alloys. Like osmium, the hardening influence of ruthenium on platinum is more than twice that of iridium, while the toxic properties of ruthenium oxide are very much like those of the osmium tetroxide; however, its rate of volatilisation is not nearly as high as that of the latter oxide, and while ruthenium-rich platinum alloys will exhibit a certain loss of weight on being heated to high temperatures, this loss becomes almost negligible with comparatively small ruthenium contents. An 8 per cent. platinum-ruthenium alloy, the hardness of which corresponds to that of a 20 per cent. platinum-iridium alloy, loses only 0.026 per cent. by weight within one hour of annealing. Small amounts of ruthenium are being employed, together with iridium, as a hardening agent in platinum alloys. Instead of adding, say, 15 per cent. of iridium to attain a certain degree of hardness, an alloy consisting of 91 per cent. platinum, 5 per cent. iridium and 4 per cent. ruthenium may be used with advantage. While the platinum-rhodium alloys may be employed for temperatures up to 2,900° F., iridium-ruthenium alloys with 10 per cent. ruthenium are serviceable for temperatures up to, and above, 3,800° F.

National Physical Laboratory Metallurgical Researches

Annual Report for the Year 1931

THE report of the Director of the National Physical Laboratory for the year 1931 (H. M. Stationery Office, price 15s. net) makes extended reference to metallurgical researches which have been carried out as part of the laboratory's programme. Dealing with the effect of surface conditions on the strength of spring steels, it is stated that previous work at the laboratory showed that the strength of spring steels when subjected to alternating or repeated stresses is largely dependent on the surface condition of the material, and that in normal unpolished spring steels only about one half the real strength of the material can be utilised owing to defective surfaces. Recent experiments, however, have determined one important cause of surface weakness, namely the production of a thin soft layer of metal at the surface during heat treatment, and improved methods of heat treatment have been developed which prevent the formation of this soft surface layer.

X-Rays in Metallurgical Research

By methods of X-ray crystal analysis it is now possible to determine the way in which the atoms of crystals are arranged, even when the crystals, as in the case of most metals, are too small to be examined microscopically. The X-ray spectra of different crystals are as a rule easily distinguishable, so that X-ray examination will often settle a question as to whether a given material consists of one or more different crystalline species. An example of the use of this method is afforded by the research which is reported on cadmium. Strips of this metal prepared by rolling differ in strength according to whether the rolling is carried out with the metal hot or cold, but an X-ray examination of the material in the hot-rolled and cold-rolled condition shows that the crystal structure is the same in both cases, but that in the cold-rolled metal the small crystals tend to set themselves with a certain layer of atoms parallel to the sheet, while in the hot-rolled state the crystals have a random orientation.

Since the war the National Physical Laboratory has carried out important pioneer work on light alloys, consisting of a light basis metal, *e.g.*, aluminium or magnesium, with minor amounts of other metals. These materials, combining lightness with strength, occupy an increasingly important position in engineering practice, and their applications are steadily extending. The results of the research work in progress at the laboratory and elsewhere, and the courage and foresight of manufacturers in applying these results in practice, have given rise to a new industry. In aircraft, these light alloys, in conjunction with special steels, have largely ousted wood for chassis and wing construction, while 50 per cent. by weight and more by volume of the engine commonly consists of aluminium alloy. An interesting new application of aluminium alloy in railway engineering is to the coupling rods of locomotive driving wheels. The laboratory has also devoted attention to processes for further improving the properties and stability of existing light metal alloys, and the present report refers to investigations on permanence of shape, on processes for removing gaseous impurities which cause unsoundness in castings and ingots, and on the mechanism of the hardening which certain of these alloys undergo spontaneously at room temperature after quenching in water or oil from a black heat, and on which their valuable properties largely depend. In its work on magnesium alloys the laboratory is searching for stronger alloys than are at present available. Magnesium, which is 40 per cent. lighter than aluminium (itself 60 per cent. lighter than steel) is handicapped by its comparatively ready corrodibility, and its inflammability when molten. Considerable advances have, however, been made in overcoming both these difficulties, and magnesium alloys to-day can be melted and cast in the foundry almost as easily as aluminium, while efficient processes have been developed for affording protection from corrosion.

Ductile Beryllium

Considerable attention has recently been devoted to a study of the new light metal beryllium. Whereas 10 years ago beryllium was a scientific curiosity, successful methods of production have been rapidly evolved, as a result of intensive research both in this country, at the National Physical Laboratory, and in Germany. The metal, which resembles steel in colour, is considerably lighter than aluminium, but possesses a very much higher melting point. Recent work at the laboratory has therefore been directed

towards the production of pure metal, free from the impurities which are normally present in the commercial product and which render it very brittle. In spite of many difficulties, pure beryllium has now been obtained, and has been found to be ductile and strong.

A series of tests has been carried out for the Aeronautical Research Committee to ascertain whether additions of "dopes" such as lead ethyl improve the properties of lubricating oils. At present the maximum temperature permitted in the oil supply to aircraft and motor-car engines is about 70° C., and it was hoped that the safe running temperature might be considerably raised by the addition of compounds such as lead ethyl. Although this has not been proved by the experiments, it has been found that if the bearings have a good surface finish, temperatures of the order of 200° C. may be reached with apparent safety. Evidence has been obtained that the use of castor-oil containing lead ethyl for the preliminary running-in of the engine bearings may produce the desired surface finish. The results of this work indicate that the lubricating oils in present use may be run safely at much higher temperatures than have hitherto been considered desirable, with corresponding reduction in friction losses, or alternativey, that oils of lower viscosity than those now used may be employed, with corresponding reduction of the starting friction when cold.

Some Inventions of the Month

Corrosion-Resistant Iron

ACCORDING to Specification No. 366,511, by Electro Metallurgical Co., of 30 East 42nd Street, New York, a corrosion-resistant iron alloy contains from 16 to preferably not more than 22 per cent. of chromium, from 6 to preferably not more than 16 per cent. of manganese, 1.5-14 per cent. of cobalt, and preferably not more than 0.3 per cent. of carbon. An example contains 18 per cent. of chromium, 8 per cent. of manganese, 5 per cent. of cobalt and 0.12 per cent. of carbon.

Non-Magnetic Steels

A NON-MAGNETIC steel alloy contains 16.25 per cent. of manganese, 0.1-0.4 per cent. of carbon, and up to 1.5 per cent. of silicon, with or without up to 5 per cent. of one or more other elements such as titanium, chromium, vanadium and molybdenum, is claimed in Specification No. 366,326, by F. Krupp, Akt.-Ges., of Essen, Germany. The alloy described may have a wholly or mainly austenitic structure produced by heat-treatment such as quenching from about 1,000° C. or by hot shaping, such as casting, rolling or forging.

Titanium in Aluminium-Silicon Alloys

IN Specification No. 366,654 by Aluminium, Ltd., of 22 Canada Life Building, Toronto, there is described an aluminium-silicon alloy for casting, which contains about 3.15 per cent. of silicon, up to .05 per cent. of titanium, and the balance aluminium. This alloy may be made by adding titanium in the course of the electrolytic production of aluminium, for example, by adding titanium oxide or titaniferous alumina to the fluoride bath, so that the reduced titanium will alloy with the aluminium, silicon being afterwards added as such or as an aluminium-silicon alloy.

Alloys for High Temperature

APPARATUS for carrying out reactions at elevated temperature in presence of hydrogen is made of or lined with an alloy containing titanium, iron and aluminium, and optionally also one or more of the elements chromium, tungsten, molybdenum, vanadium, silicon, nickel, cobalt, copper, manganese, tin, zinc, lead, silver and beryllium, according to claims put forward in Specification No. 366,762, of J. Y. Johnson (a communication from I. G. Farbenindustrie). Specified alloys are (1) iron 92.5 per cent, aluminium 6 per cent., titanium 1.5 per cent.; (2) iron 91 per cent., aluminium 5 per cent., chromium 3 per cent., titanium 1 per cent.

Nitrogenising Iron Alloys

THE nitrogenisation of alloys of iron, steel and cast iron carried out in the presence of one or more halogen compounds, particularly chlorine compounds, is described in Specification No 366,838 by F. Krupp Akt.-Ges., of Essen, Germany. In an example, a steel containing 0.3 per cent. of carbon, 0.2 of silicon, 0.3 of manganese, 1.5 of chromium and 1.2 of aluminium is sprinkled with calcium chloride and nitrogenised for 48 hours at 500° C. Ammonium chloride and bleaching powder may also be used. The alloys treated contain one or more of the metals aluminium, chromium, molybdenum, vanadium and titanium and further elements such as silicon, manganese, nickel and tungsten may be incorporated.

Addition of Lead to Aluminium

LEAD is incorporated in aluminium by means of magnesium so as to produce a ternary alloy containing about 2 per cent. of lead in the form of a homogeneous solid solution, according to Specification No. 366,483, of Soc. des Brevets B. de Montby, 2 Rue Scribe, Paris. The aluminium is melted in a plumbago crucible lined with magnesia and the bath is cleaned by adding deoxidising mixtures such as mixtures of calcium fluoride and sodium fluoride. In one example, a magnesium-lead alloy is then added of such a composition that the final alloy contains 0.8-1 per cent. of magnesium. After further cleaning with the deoxidising mixture the alloy is tapped. Alternatively the magnesium and lead may be added in the unalloyed form.

Aluminium Alloys

ALUMINIUM and aluminium alloys are improved by introducing into the molten metal one or more of the chlorides of silicon, carbon, titanium or aluminium, according to Specification No. 366,545 of W. Rosenhain and J. D. Grogan, of the National Physical Laboratory, Teddington. The chlorides may be passed into the metal contained in a crucible, whilst large quantities of metal may be treated in a vessel resembling a Bessemer converter. Solid aluminium chloride may be placed on the surface of the metal and forced down by a plunger or other device. The chlorides may also be volatilised into a stream of inert gas, *e.g.* nitrogen, argon, helium, hydrocarbons, or other halogen compounds, and be carried by the stream into the metal. Titanium may be introduced into the metal when titanium chloride is used. Amongst other effects produced the treatment according to the invention reduces the grain size in the metal.

Extracting Metals from Slags

ACCORDING to Specification No. 366,168, by M. Lissauer, H. Lissauer and B. Griesmann, of 17 Elisenstrasse, Cologne, non-ferrous metals such as copper, nickel, etc., and also more volatile metals such as lead, tin, antimony, etc., are recovered wholly or partly in reguline form from molten slags to which may also be added substances containing metal oxides such as flue dust, poor ores, etc., by passing a reducing gas, preheated if desired, through the melt, while maintaining a neutral or reducing atmosphere over the melt by adjustment of the nature of the combustion gases and the amount of reducing gas employed. As the bath becomes poor in metal content an oxidising atmosphere may be maintained above the bath in order to volatilise the residual metals as oxides and the volatilisation may be accelerated by the addition of sulphur or sulphur bearing substances to the melt when volatile sulphur compounds will be driven out and may be burned to oxides or sulphates.

Nickel Steel Plate Manufacture

THE increasing use of alloy steels in general engineering is an interesting sign of the desire to take advantage of the higher stresses for which these materials are useful, whilst at the same time reducing weight. Keeping pace with this trend the Appleby (Lincs) works of The United Steel Companies, Ltd., which has previously manufactured only mild and medium carbon steels for boiler, ship, bridge, tank and similar structural purposes, is now manufacturing nickel steel plates in all sizes from $\frac{1}{8}$ in. thick upwards.

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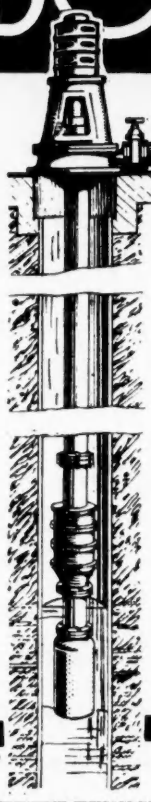

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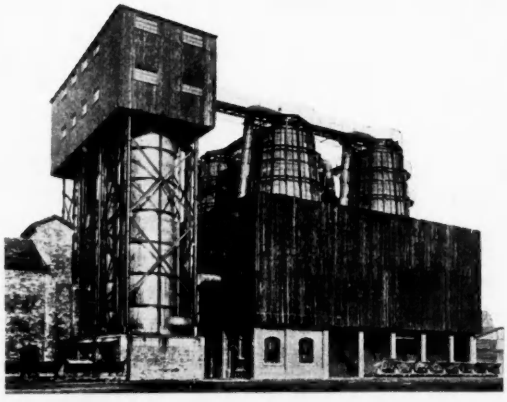

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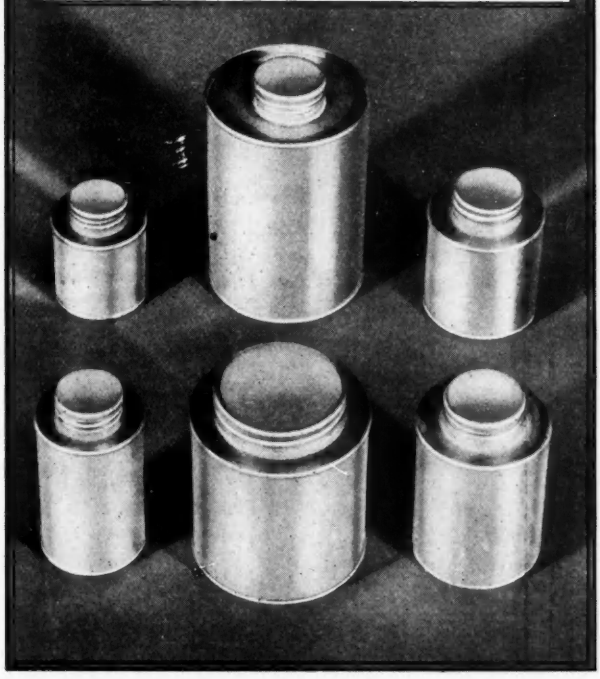

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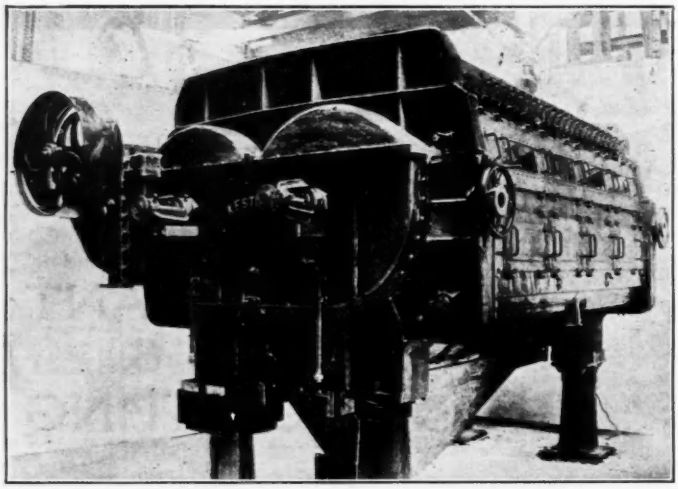

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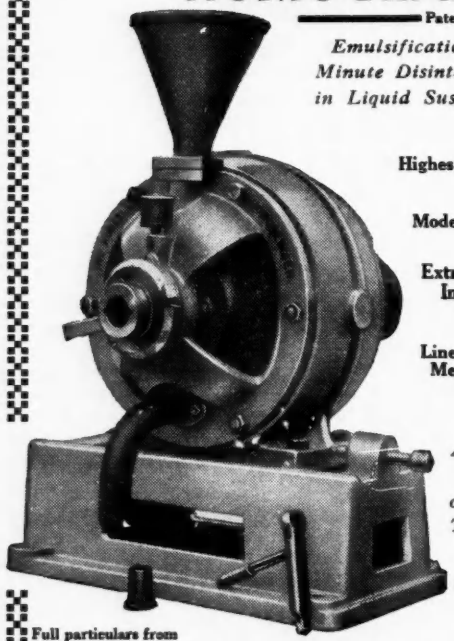

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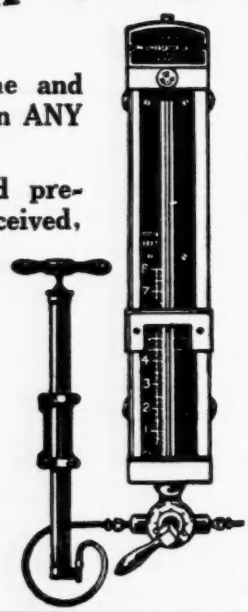

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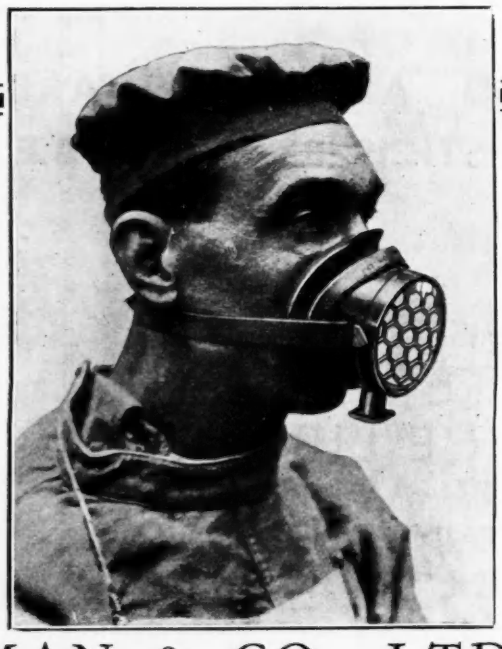

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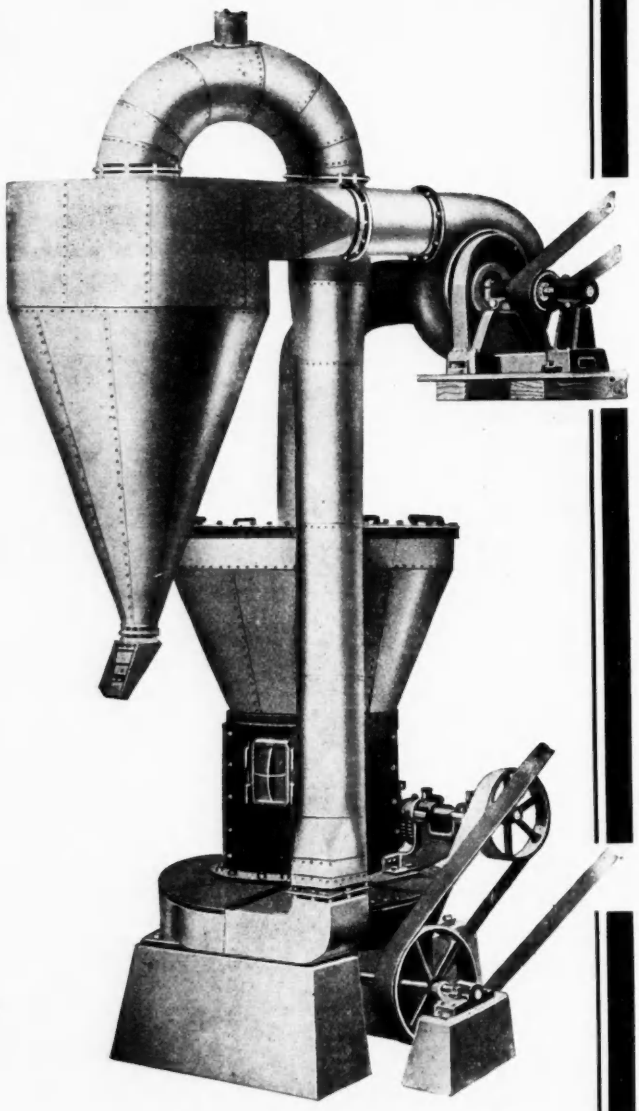

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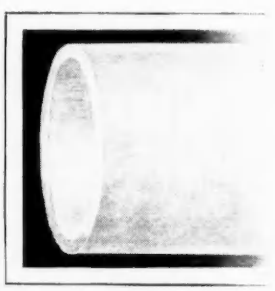

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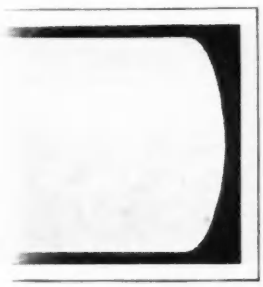

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The following are taken from printed reports, but we cannot be responsible for any errors that may occur.

Mortgages and Charges

[NOTE.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described therein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every Company shall, in making its Annual Summary, specify the total amount of debts due from the Company in respect of all Mortgages or Charges. The following Mortgages and Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an *—followed by the date of the Summary, but such total may have been reduced.]

CEMENT INDUSTRIES, LTD., London, S.W. (M., 25/6/32.) Registered June 9. £1,600 debenture, part of £70,000; general charge.

EASTERN CHEMICAL CO., LTD., London, E.C. (M., 25/6/32.) Registered June 8, charge, supplemental to charge dated December 14, 1927, securing cash advances made and to be made by charges, E. D. Sassoon and Co., Ltd., Bombay, to or on account of company and not otherwise secured, not exceeding 500,000 rupees, instead of former amount of 200,000 rupees; charged on company's works and premises at Matunga, Bombay. *£30,100. December 2, 1931.

Satisfaction

POLYSULPHIN CO., LTD. (old company), Keynsham, chemical manufacturers, &c. (M.S., 25/6/32.) Satisfaction registered June 10, of debentures registered February 1, 1926, to extent of £9,000.

County Court Judgments

[NOTE.—The publication of extracts from the "Registry of County Court Judgments" does not imply inability to pay on the part of the persons named. Many of the judgments may have been settled between the parties or paid. Registered judgments are not necessarily for debts. They may be for damages or otherwise, and the result of bona-fide contested actions. But the Registry makes no distinction of the cases. Judgments are not returned to the Registry if satisfied in the Court books within twenty-one days. When a debtor has made arrangements with his creditors we do not report subsequent County Court judgments against him.]

HUNT, FRANK LEWIS, Babcary, near Yeovil, chemical engineer. (C.C., 25/6/32.) £20 7s. 2d. April 11.

MARRIOTT, P. J. (male), 136 Walnut Street, Leicester, chemical manufacturer. (C.C. 25/6/32.) £14 1s. 9d. April 22.

Chemical Trade Inquiries

Abstracted from the "Board of Trade Journal." Names and addresses may be obtained from the Department of Overseas Trade (Development and Intelligence), 35 Old Queen Street, London, S.W.1 (quote reference number).

Brazil.—A firm in Sao Paulo wishes to obtain the representation of United Kingdom manufacturers of chemical products for the textile industry. (Ref. No. 1072.)

Japan.—A British subject resident in Japan desires to obtain the agencies of United Kingdom manufacturers of industrial and pharmaceutical chemicals, metals, structural steel, etc. (Ref. No. 1068.)

Canada.—A manufacturers' agent at Toronto desires to obtain United Kingdom agencies in lines selling to the drug trade, preferably on a commission basis, for the whole Dominion. (Ref. No. 1054.)

Canada.—A newly-established firm of manufacturers' agents in Toronto desires to represent United Kingdom manufacturers of feeds and fertilisers, gelatine, vegetable oils, greases and tallows, on a commission basis, in the Province of Ontario. (Ref. No. 1052.)

Egypt.—The Egyptian Ministry of Agriculture is calling for tenders to be presented in Cairo by June 30, for the supply of 56½ tons of commercial sulphuric acid, and 25 tons of sodium cyanide for the year 1932-1933. Particulars have been communicated to firms whose names are on the special register. (Ref. No. F.X.1522.)

British India.—The Director-General, India Store Department, Belvedere Road, Lambeth, London, S.E.1, invites tenders for 40 tons caustic soda and 10 tons soda ash. Samples required with tender. Tenders due July 5. Forms of tender obtainable from the Director-General at a fee of 5s. which is not returnable.

British India.—The Director-General, India Store Department, Belvedere Road, Lambeth, London, S.E.1, invites tenders for (Schedule 1) 6,600 dozen bottles, glass, 1 oz. to 4 lb.; (2) 39,530 lb. lint, plain; (3) 24,500 gal. cresol saponified; (4) iodum and iodoformum B.P., and (5) Bismuthi subnitras, carbonas, etc., B.P. Samples required with tender for schedules 1, 2 and 3. Tenders due July 5, for schedule 1 and July 8 for schedules 2, 3, 4 and 5. Forms of tender obtainable from the Director-General at a fee (which will not be returned) of 5s. for each schedule.

Other Inquiries

The following inquiries have also been received. Except where otherwise stated, information should be sent to the Editor of The Chemical Age.

Sulphite Cellulose Waste.—A firm of chemical manufacturers is anxious to obtain supplies of lignin sulphonic acid (sulphite cellulose waste), a by-product from the manufacture of viscose silk. (Ref. No. C.A. 181.)

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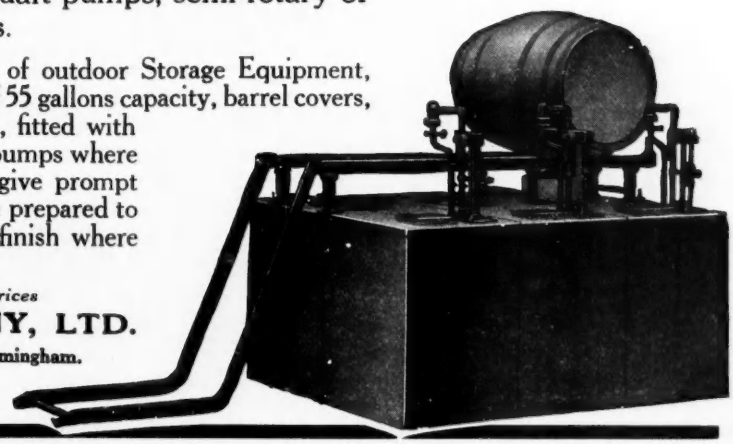

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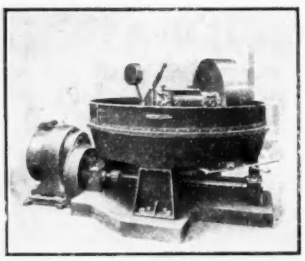

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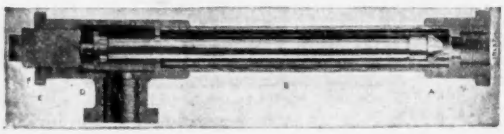

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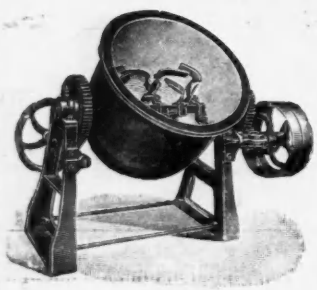

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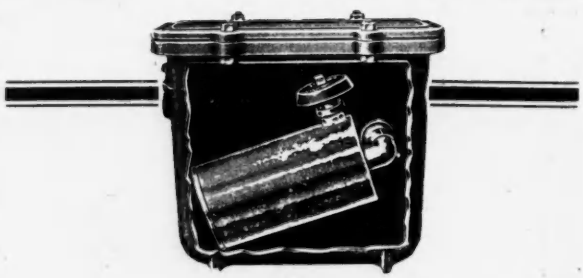

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